AD-A185 462 X-RAY STRUCTURE ANALYSES AND MOLECULAR MMCHANICS ANALYSES OF DENSE ENERGETIC MATERIALS(U) NAVAL RESEARCH LAB WASHINGTON DC R GILARDI ET AL. 01 JUL 87 1/2

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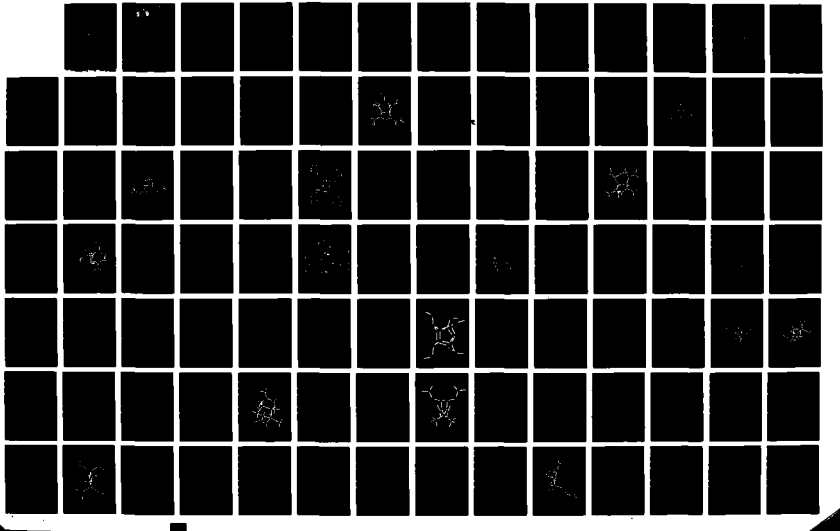

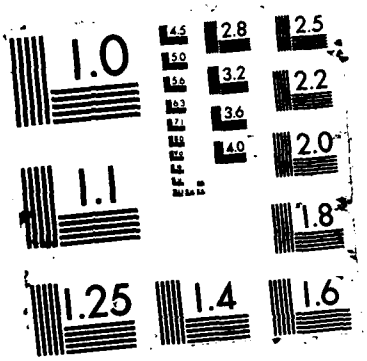
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REPORT DOCUMENTATION PAGE

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17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB-GROUP	X-ray Diffraction, nitramines, hexaazaisowurtzitane

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

Structural characterization of 22 energetic materials and precursors was performed with X-ray diffraction. The materials examined include: (Pls. see reverse side)

20. DISTRIBUTION / AVAILABILITY OF ABSTRACT ☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT. ☐ DTIC USERS	21. ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED
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SECURITY CLASSIFICATION OF THIS PAGE

a 2,4-Dinitro-6,8-diacetyl-2,4,6,8-tetraazabicyclo[3.3.0]octane

b 2,5-Dinitro-7,9-diacetyl-2,5,7,9-tetraazabicyclo[4.3.0]nonane

c 2,4,6-Trinitro-8,10-diacetyl-2,4,6,8,10-pentaazabicyclo[5.3.0]decane

d 2,4-Dinitro-6,8,-dipropionyl-2,4,6,8-tetraazabicyclo[3.3.0]octane

e 2,4-Diacetyl-6-nitro-2,4,6-triaza-8-oxabicyclo[3.3.0]octane

f 2,4,6,8-Tetrabenzyl-2,4,6,8-tetraazabicyclo[3.3.0]octane

g 2,5,7,9-Tetranitro-8-acetoxy-2,5,7,9-tetraazabicyclo[4.3.0]nonane

h 1,4,6,9-Tetranitro-1,4,6,9-tetraaza-5,10-dioxaperhydroanthracene

i 2-Oxa-6,9-diaza-6,9-dinitrospiro[3.6]decane

j 8,11-Dibromo-8,11-dinitro-pentacyclo[5.4.0.$0^{2,6}$.$0^{3,10}$.$0^{5,9}$]undecane

k 8-Bromo-8-nitro-pentacyclo[5.4.0.$0^{2,6}$.$0^{3,10}$.$0^{5,9}$]undecane-11-one

l 6-Dihydroxy-2,7-diethoxycarbonyl pentacyclo[6.5.0.$0^{1,8}$.$0^{4,5}$.$0^{9,13}$]tri-deca-2,6-diene

m 2,4,6,8,10,12-Hexa(4-methoxybenzyl)-2,4,6,8,10,12-hexaazatetracyclo-[5.5.0.$3^{3,11}$.$0^{5,9}$]dodecane

n 2,3,4,8-Tetraphenyl-6,6-anti-10-trinitropentacyclo[5.3.0.$0^{2,5}$.$0^{3,9}$.$0^{4,8}$]decane

o 1,6-Dimethyl-1α-4α,4aα,5α 8β,8aα-hexahydro-1,4-methanonaphthalene

p 2,2-Bis(trifluoromethyl)-4,5-diacetoxy-1,3-diacetyl-imidazolidine

q 2-Acetoxy-3(N-acetamidomethyl-N-nitroso)amino-1,4,-dinitropiperazine

r 1,1,3,3-Tetranitrocyclobutane

s Bimethylene trisacetamide

t 5H,10H-di-tetrazolo[1,5-a;1,5-d]piperazine

u 1,4-Dinitro-2-acetoxy-3-hydroxy-1,4-diazacyclohexane

v 1,4-Butanediammonium dinitrate

X-ray Structure Analyses and Molecular Mechanics Analyses of Dense Energetic Materials

by Richard Gilardi , Clifford George and Judith Flippen-Anderson
Naval Research Laboratory, Washington, D.C. 20375

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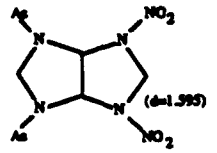

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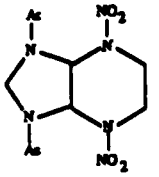

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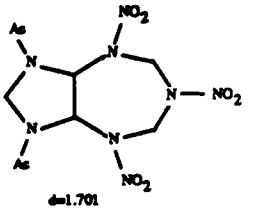

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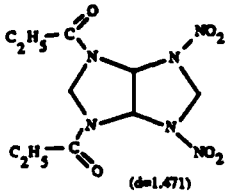

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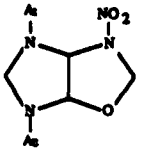

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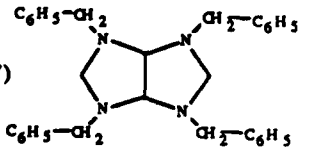

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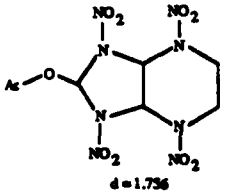

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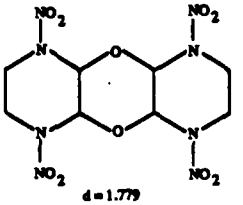

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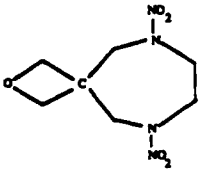

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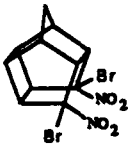

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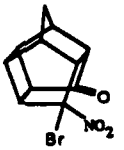

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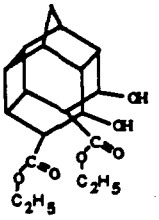

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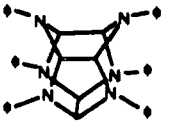

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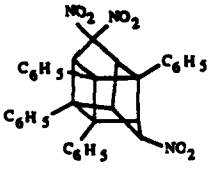

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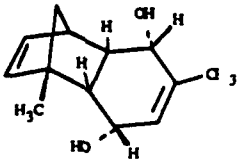

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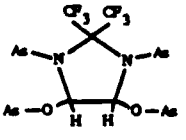

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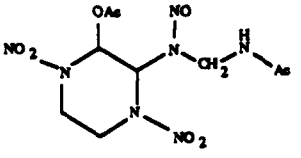

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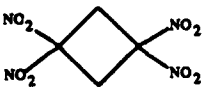

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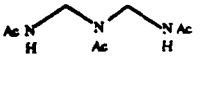

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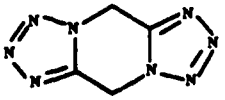

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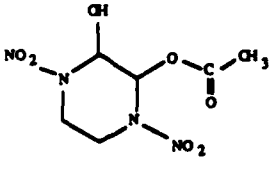

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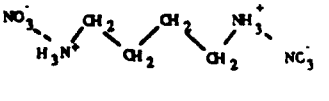

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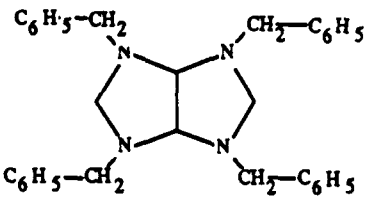
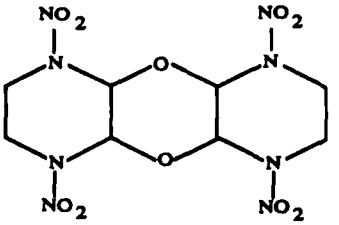
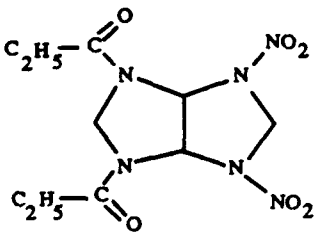
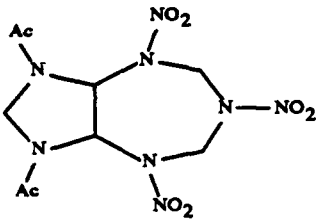
Introduction

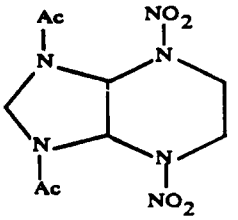
More than thirty energetic compounds were studied with X-rays at NRL in 85/86, and several seem to be very close precursors to long-standing target molecules or target classes of compounds. In many cases, X-ray diffraction analyses can provide detailed structures even when the structural and empirical formulae for the compounds are unknown. This is particularly important when material is very scarce, or when the solution spectra are complicated by complex conformational equlibria. Several "unknown nitrolysis products" were determined at NRL this year. The group of molecular structures pictured below were determined at NRL, and are all fused-ring nitramines of the bicyclo-HMX type. The compounds in the first row below come from NWC (China Lake, CA) and NSWC -WO (Silver Spring, MD). All of the others on this page were made at the Lawrence Livermore Lab (Livermore, CA).

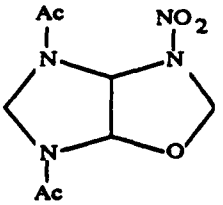


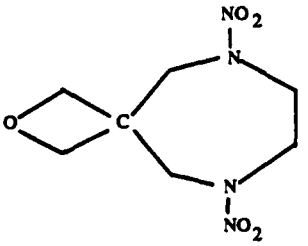


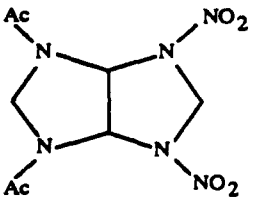


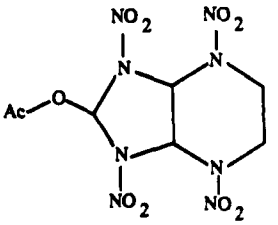


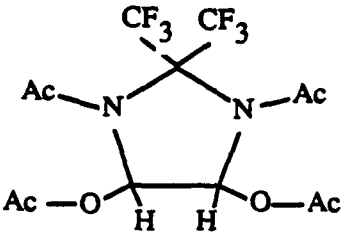


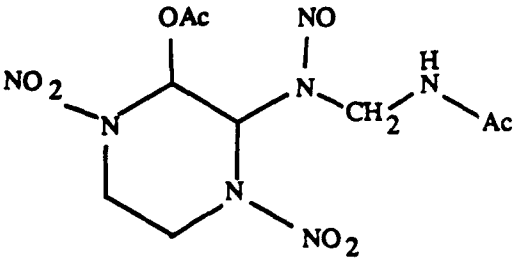


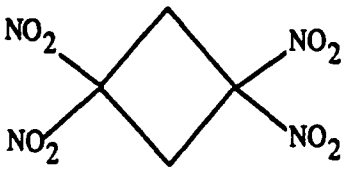


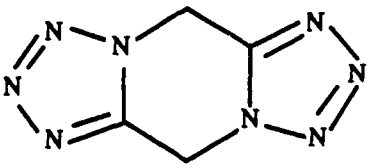


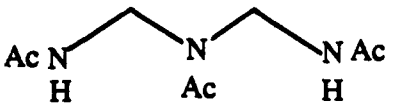


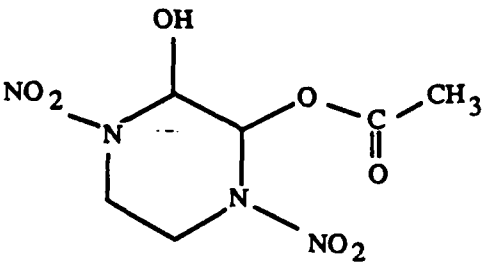


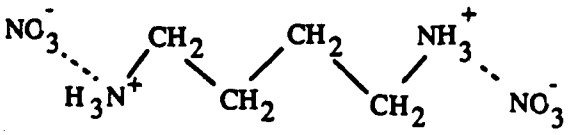






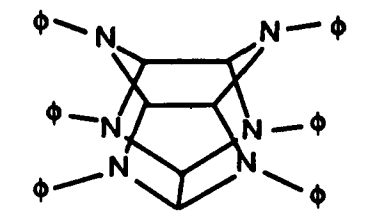




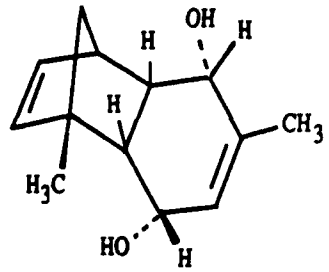


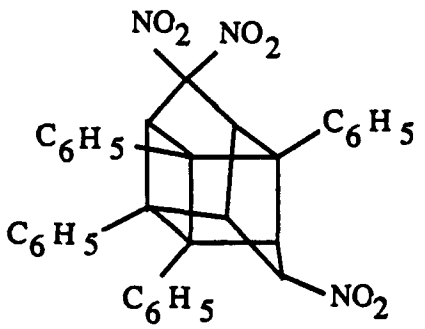
The remaining compounds come from a variety of laboratories; several are primarily of theoretical interest, and two were unknown reaction products which were identified by their diffraction. However, tetranitrocyclobutane (second row), made by Dr. K. Baum (Fluorochem; Azusa, Calif.) is a dense strained energetic material which may be a useful formulant. The final compound, a butanediammonium dinitrate, is a currently used propellant formulant. Its decomposition mechanism, and its relation to molecular structure and crystal packing, is being studied by Dr. R. McKenney (Eglin AFB).

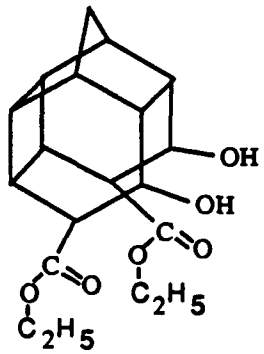
The remainder of this report consists of a series of summary reports on the individual molecular structures. These summaries each follow the same format. First, a brief report of the crystal data and conformational data which serve to uniquely identify the particular material studied. Second, a description of the X-ray experiment and the results of the analysis and refinement of the structure. Third, a picture of the molecule, and finally, tables of fractional unit cell coordinates, bond distances, and bond angles. From this data, one should be able to calculate any details of the molecular structure or crystal packing not included in the summary.

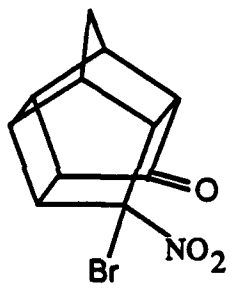


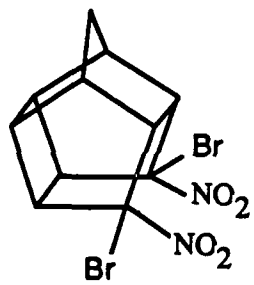
Φ = $CH_2-C_6H_5-OCH_3$











The compounds pictured above were received from researchers involved in the synthesis of new nitro or nitramino cage compounds. Early in 1986, Nielsen (NWC, China Lake) achieved the synthesis of hexa-azaisowurtzitane via a glyoxal-benzylamine condensation (top left molecule); in October, the dinitro, tetraacetyl derivative (top right) was made and corroborated by X-ray analysis. This is the first nitro-aza cage ever reported, a possible precursor to the hexanitro target, and a good model for theoretical studies of the target compound.

The remaining compounds pictured above come from Drs. Marchand (N. Texas State U.) and Zajac (Villanova), who are developing methods for the synthesis of carbon-based cages and their polynitro substitution. In these materials, a sizeable amount of energy is stored in the cage itself, primarily as angle-bending strain. Molecular mechanics calculations can pin-point the locations of highest strain for hypothetical (or actual) cage molecules; molecular mechanics analyses on several cage compounds have been completed and are, or will be described in the literature. In general, a comparison of the results with crystal structure findings reveals a close correspondence; bond angles are predicted to within a few degrees, and bond distance deviations from normality caused by steric strain are qualitatively predicted by the theory, but are often found to be larger, experimentally, than predicted.

Abstract

2,4-Dinitro-6,8-diacetyl-2,4,6,8-tetraazabicyclo[3.3.0]octane, $C_8H_{12}N_6O_6$, M_r = 288.22 , orthorhombic $P2_12_12_1$, a = 8.480(2), b = 11.845(2), c = 11.944(2) Å, V = 1199.7(3) Å^3, Z = 4, D_x = 1.595 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 10.89 cm^{-1}, F(000) = 600 , T = 295 K, Final R = 0.036, wR = 0.049, for 1052 independent reflections. All four ring nitrogens are directed away from the cleft of the two rings. The two ring nitrogens involved in N-N bonding are pyramidal, with angles between the exocylic N-N bonds and the C-N-C planes of 44.4 and 42.5° .

Experimental

A clear colorless 0.27 x 0.24 x 0.20 mm data crystal was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within $39 \leq 2\theta \leq 77°$ used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.57 Å^{-1}, range of hkl : $0 \leq h \leq 9$, $-13 \leq k \leq 0$, $0 \leq l \leq 13$. Standards 400, 333, 006, monitored every 60 reflections with random variation of 2.0 % over data collection, θ/2θ mode, scan width $(2.0 + \Delta_{\alpha 1 \alpha 2})°$, scan rate a function of count rate (5.0 °/min. minimum, 30.0° /min. maximum), 2497 reflections measured, 2011 unique, R_{int} = 0.04, 1307 observed with $Fo > 3\sigma(Fo)$. Data corrected for Lorentz and polarization, but not absorbtion effects . Structure solved by direct methods. The least-squares refinement used the MicroVAX version of the SHELXTL system (Sheldrick, 1980). $\Sigma w(|Fo|-|Fc|)^2$ minimized where $w = 1/[\sigma^2(|Fo|) + g.(Fo)^2]$, g = 0.00025. 221 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model , C-H = 0.96 Å, C-H-C = 109.5° , U(H) = 1.1· U_{eq}(C) . $(\Delta/\sigma)_{max}$ = 0.085, R = 0.075, wR = 0.064, S = 1.44. Final difference Fourier excursions 0.34 and -0.32 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, and 2, atom coordinates, bond distances and angles, follows that shown in Fig. (1a).

2,4-Dinitro-6,8-diacetyl-2,4,6,8-tetraazabicyclo[3.3.0]octane

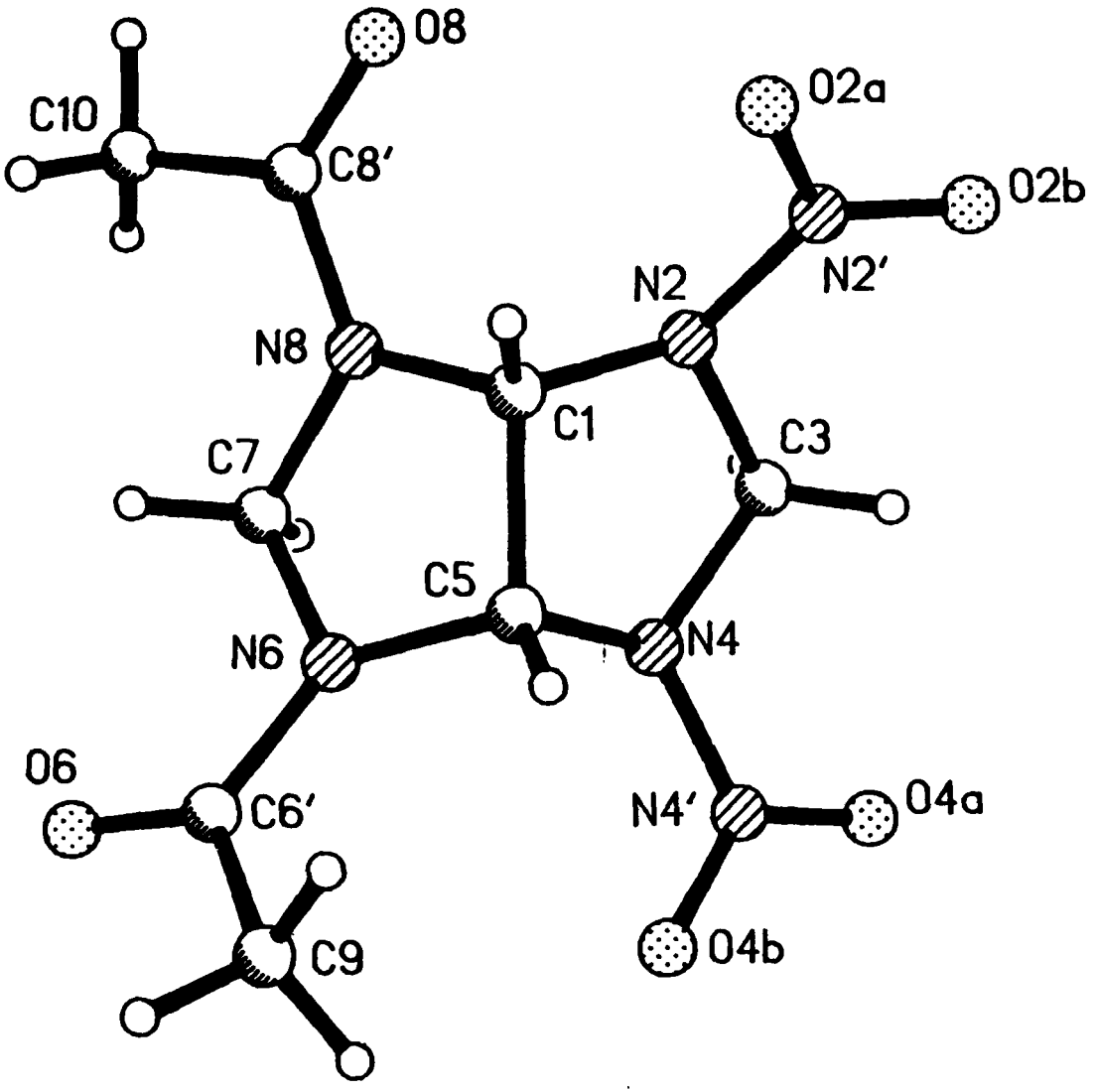


Table 1a. Atom coordinates ($x10^4$) and thermal parameters ($Å^2 \times 10^3$)

atom	x	y	z	U
C(1)	7329(3)	2534(2)	3636(2)	35(1)*
N(2)	8668(3)	2015(2)	3088(2)	42(1)*
C(3)	8538(3)	2002(3)	1880(3)	51(1)*
N(4)	6902(3)	2373(2)	1713(2)	40(1)*
C(5)	6002(3)	2534(2)	2750(2)	35(1)*
N(6)	5332(3)	3648(2)	2819(2)	38(1)*
C(7)	6527(3)	4461(2)	3186(3)	42(1)*
N(8)	7572(3)	3733(2)	3847(2)	39(1)*
N(2')	9391(3)	1089(2)	3571(3)	57(1)*
O(2a)	9124(3)	904(2)	4555(2)	79(1)*
O(2b)	10325(3)	571(2)	2977(3)	81(1)*
N(4')	6153(3)	1946(2)	803(2)	47(1)*
O(4a)	6976(3)	1582(2)	48(2)	70(1)*
O(4b)	4728(3)	2019(2)	788(2)	59(1)*
C(6')	3789(3)	3990(2)	2733(2)	41(1)*
O(6)	3506(2)	5004(2)	2735(2)	55(1)*
C(9)	2534(4)	3104(3)	2653(3)	53(1)*
C(8')	8523(3)	4088(3)	4698(3)	45(1)*
O(8)	9272(3)	3404(2)	5244(2)	67(1)*
C(10)	8581(4)	5332(3)	4903(3)	57(1)*

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2a. Bond Lengths (Å) and bond angles (deg.)

C(1)-N(2)	1.448(3)	C(1)-C(5)	1.546(4)
C(1)-N(8)	1.456(3)	N(2)-C(3)	1.447(4)
N(2)-N(2')	1.383(4)	C(3)-N(4)	1.469(4)
N(4)-C(5)	1.467(4)	N(4)-N(4')	1.356(3)
C(5)-N(6)	1.438(3)	N(6)-C(7)	1.465(3)
N(6)-C(6')	1.374(3)	C(7)-N(8)	1.467(4)
N(8)-C(8')	1.364(4)	N(2')-O(2a)	1.216(4)
N(2')-O(2b)	1.228(4)	N(4')-O(4a)	1.219(4)
N(4')-O(4b)	1.212(4)	C(6')-O(6)	1.225(3)
C(6')-C(9)	1.498(4)	C(8')-O(8)	1.218(4)
C(8')-C(10)	1.495(4)		

N(2)-C(1)-C(5)	105.2(2)	N(2)-C(1)-N(8)	112.4(2)
C(5)-C(1)-N(8)	102.8(2)	C(1)-N(2)-C(3)	113.3(2)
C(1)-N(2)-N(2')	119.8(2)	C(3)-N(2)-N(2')	116.2(2)
N(2)-C(3)-N(4)	101.8(2)	C(3)-N(4)-C(5)	114.6(2)
C(3)-N(4)-N(4')	116.1(2)	C(5)-N(4)-N(4')	118.7(2)
C(1)-C(5)-N(4)	101.5(2)	C(1)-C(5)-N(6)	104.4(2)
N(4)-C(5)-N(6)	112.0(2)	C(5)-N(6)-C(7)	110.3(2)
C(5)-N(6)-C(6')	129.9(2)	C(7)-N(6)-C(6')	119.2(2)
N(6)-C(7)-N(8)	101.1(2)	C(1)-N(8)-C(7)	113.3(2)
C(1)-N(8)-C(8')	120.8(2)	C(7)-N(8)-C(8')	125.2(2)
N(2)-N(2')-O(2a)	117.6(3)	N(2)-N(2')-O(2b)	116.2(3)
O(2a)-N(2')-O(2b)	126.1(3)	N(4)-N(4')-O(4a)	117.1(3)
N(4)-N(4')-O(4b)	116.9(2)	O(4a)-N(4')-O(4b)	125.8(3)
N(6)-C(6')-O(6)	118.4(2)	N(6)-C(6')-C(9)	118.4(2)
O(6)-C(6')-C(9)	123.2(3)	N(8)-C(8')-O(8)	120.1(3)
N(8)-C(8')-C(10)	116.5(3)	O(8)-C(8')-C(10)	123.4(3)

Abstract

2,5-Dinitro-7,9-diacetyl-2,5,7,9-tetraazabicyclo[4.3.0]nonane, $C_9H_{14}N_6O_6$• 1/2(C_3H_6O), M_r = 331.29, monoclinic, C2/c, a = 29.512(5), b = 9.142(1), c = 10.872(2) Å, β = 92.46(1)°, V = 2930.6(9) $Å^3$, Z = 8 D_x = 1.501 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 9.61 cm^{-1}, F(000) = 1392, T = 295 K, Final R = 0.043, wR = 0.047 for 1311 independent reflections.The compound crystallizes with 1/2 molecule of acetone per asymmetric unit. The alkyl substituted five membered ring is a flattened envelope with N(7) the out of plane atom. The six membered ring has a boat conformation with the two N atoms out of the plane formed by its four C atoms. One nitramine group is slightly pyramidal (C-N bending angle = 17.1°) and the other is pyramidal (C-N bending angle = 38.5°).

Experimental

A clear colorless 0.30 x 0.32 x 0.07 mm data crystal was provided by C. Coon of Lawrence Livermore Laboratories. Automated Nicolet R3mμ diffractometer with incident beam monochromator. 25 centered reflections within $30 \leq 2\theta \leq 59°$ used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.55 $Å^{-1}$, range of hkl : $-32 \leq h \leq 8$, $-10 \leq k \leq 0$, $11 \leq l \leq 11$. Standards 023, 040, 10 0 0 , monitored every 60 reflections with random variation of 2.5 % over data collection, θ/2θ mode, scan width [2θ($K\alpha_1$) - 0.9] to [2θ($K\alpha_2$) + 0.9]°, scan rate a function of count rate (6.0°/min. minimum, 30.0°/min. maximum), 2953 reflections measured, 1708 unique, R_{int} = 0.0[illegible], 1311 observed with $Fo > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorbtion effects. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. Secondary extinction parameter p = 0.0008(1) in $F_c^* = F_c/[\ 1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. 219 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model , C-H = 0.96 Å, C-H-C = 109.5° , U(H) = 1.2· U_{eq}(C) . (Δ/σ) max = 0.008, R = 0.043, wR = 0.047, S = 1.64. Final difference Fourier excursions 0.25 and -0.23 $eÅ^{-3}$. Atomic scattering factors from International Tables for X-

ray Crystallography (1974).* Atom numbering for tables 1, and 2, atom coordinates, bond distances and angles, follows that shown in Fig.(1b)

2,5-Dinitro-7,9-diacetyl-2,5,7,9-tetraazabicyclo[4.3.0]nonane

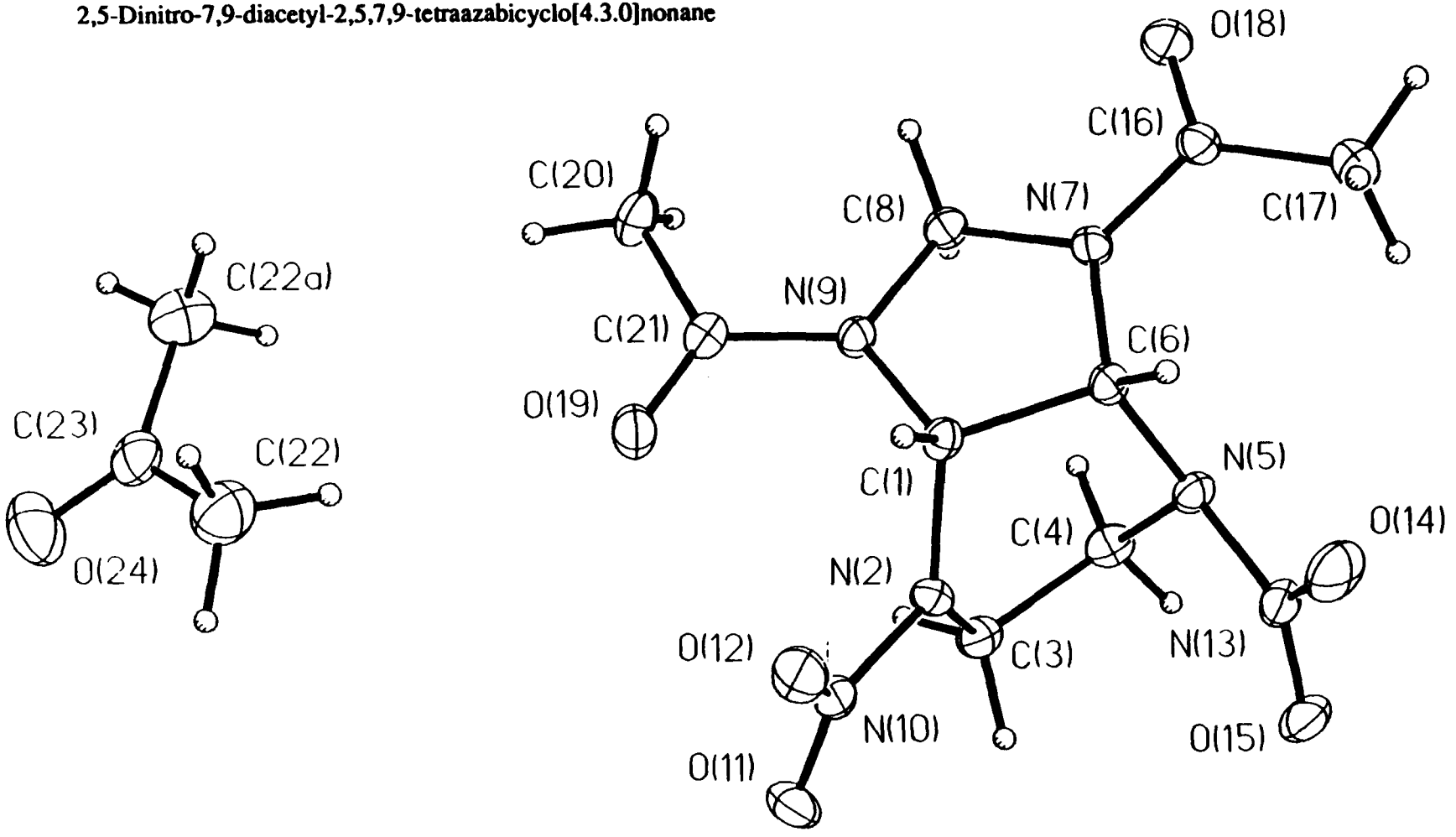


Table 1b. Atom coordinates ($x10^4$) and thermal parameters ($Å^2 \times 10^3$)

atom	x	y	z	U
C(1)	3715(1)	7169(3)	1578(2)	32(1)*
N(2)	3330(1)	6193(2)	1366(2)	35(1)*
C(3)	2981(1)	6045(3)	2256(2)	41(1)*
C(4)	2909(1)	7551(3)	2830(2)	42(1)*
N(5)	3047(1)	8723(2)	2021(2)	37(1)*
C(6)	3536(1)	8727(3)	1813(2)	33(1)*
N(7)	3788(1)	9197(2)	2906(2)	35(1)*
C(8)	4021(1)	8013(3)	3563(2)	38(1)*
N(9)	3996(1)	6831(2)	2671(2)	33(1)*
N(10)	3363(1)	5090(3)	519(2)	44(1)*
O(11)	3114(1)	4031(2)	637(2)	60(1)*
O(12)	3623(1)	5267(2)	-318(2)	54(1)*
N(13)	2766(1)	8953(2)	976(2)	44(1)*
O(14)	2919(1)	9686(2)	143(2)	67(1)*
O(15)	2381(1)	8475(2)	991(2)	56(1)*
C(16)	3816(1)	10585(3)	3353(2)	39(1)*
C(17)	3574(1)	11772(3)	2640(3)	54(1)*
O(18)	4036(1)	10811(2)	4319(2)	53(1)*
O(19)	4239(1)	4728(2)	1884(2)	51(1)*
C(20)	4595(1)	5489(3)	3797(2)	53(1)*
C(21)	4267(1)	5629(3)	2706(2)	39(1)*
O(24)	5000	-975(4)	2500	96(2)*
C(23)	5000	341(5)	2500	60(2)*
C(22)	4802(1)	1194(4)	1443(3)	83(2)*

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2b. Bond Lengths (Å) and bond angles (deg.)

C(1)-N(2)	1.453(3)	C(1)-C(6)	1.544(3)
C(1)-N(9)	1.453(3)	N(2)-C(3)	1.450(3)
N(2)-N(10)	1.372(3)	C(3)-C(4)	1.531(4)
C(4)-N(5)	1.455(3)	N(5)-C(6)	1.470(3)
N(5)-N(13)	1.394(3)	C(6)-N(7)	1.440(3)
N(7)-C(8)	1.453(3)	N(7)-C(16)	1.360(3)
C(8)-N(9)	1.451(3)	N(9)-C(21)	1.360(3)
N(10)-O(11)	1.224(3)	N(10)-O(12)	1.226(3)
N(13)-O(14)	1.229(3)	N(13)-O(15)	1.218(3)
C(16)-C(17)	1.496(4)	C(16)-O(18)	1.228(3)
O(19)-C(21)	1.216(3)	C(20)-C(21)	1.504(4)
O(24)-C(23)	1.203(6)	C(23)-C(22)	1.487(4)
C(23)-C(22a)	1.487(4)		

N(2)-C(1)-C(6)	108.8(2)	N(2)-C(1)-N(9)	114.4(2)
C(6)-C(1)-N(9)	104.4(2)	C(1)-N(2)-C(3)	121.6(2)
C(1)-N(2)-N(10)	118.8(2)	C(3)-N(2)-N(10)	116.8(2)
N(2)-C(3)-C(4)	107.6(2)	C(3)-C(4)-N(5)	111.6(2)
C(4)-N(5)-C(6)	113.4(2)	C(4)-N(5)-N(13)	115.5(2)
C(6)-N(5)-N(13)	115.4(2)	C(1)-C(6)-N(5)	111.6(2)
C(1)-C(6)-N(7)	104.1(2)	N(5)-C(6)-N(7)	110.4(2)
C(6)-N(7)-C(8)	113.6(2)	C(6)-N(7)-C(16)	126.5(2)
C(8)-N(7)-C(16)	119.9(2)	N(7)-C(8)-N(9)	102.5(2)
C(1)-N(9)-C(8)	113.5(2)	C(1)-N(9)-C(21)	120.7(2)
C(8)-N(9)-C(21)	124.8(2)	N(2)-N(10)-O(11)	116.9(2)
N(2)-N(10)-O(12)	117.8(2)	O(11)-N(10)-O(12)	125.3(2)
N(5)-N(13)-O(14)	117.3(2)	N(5)-N(13)-O(15)	117.3(2)
O(14)-N(13)-O(15)	125.3(2)	N(7)-C(16)-C(17)	118.1(2)
N(7)-C(16)-O(18)	119.0(2)	C(17)-C(16)-O(18)	122.9(2)
N(9)-C(21)-O(19)	120.3(2)	N(9)-C(21)-C(20)	116.7(2)
O(19)-C(21)-C(20)	123.0(2)	O(24)-C(23)-C(22)	121.6(2)
O(24)-C(23)-C(22a)	121.6(2)	C(22)-C(23)-C(22a)	116.7(4)

Abstract

2,4,6-Trinitro-8,10-diacetyl-2,4,6,8,10-pentaazabicyclo[5.3.0]decane, $C_9H_{14}N_8O_8$, M_r = 362.26, orthorhombic, $P2_12_12_1$, a = 8.145(1), b = 12.238(2), c = 14.186(3) Å, V = 1413.9(6) $Å^3$, Z = 4, D_x = 1.702 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 12.56 cm^{-1}, F(000) = 752, T = 295 K, Final R = 0.045, wR = 0.047 for 1325 independent reflections.

Experimental

A clear colorless 0.08 x 0.10 x 0.10 mm data crystal was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within $40 \leq 2\theta \leq 88°$ used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.59 $Å^{-1}$, range of hkl : $-9 \leq h \leq 9$, $0 \leq k \leq 12$, $0 \leq l \leq 16$. Standards 301, 042, 013, monitored every 60 reflections with linear variation of 10 % over data collection, θ/2θ mode, scan width [2θ(Kα$_1$) - 0.7] to [2θ(Kα$_2$) + 0.7]°, scan rate a function of count rate (2.0°/min. minimum, 12.0°/min. maximum), 3224 reflections measured, 1325 observed with $Fo > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorbtion effects. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. Secondary extinction parameter p = 0.0047(9) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. 257 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model (coordinate shifts of C applied to attached H atoms), C-H = 0.96 Å, C-C-H = 109.5° (methyl H), $U(H) = 1.1\ U_{eq}(C)$. (Δ/σ) max = 0.005, R = 0.045, wR = 0.047, S = 1.995. Final difference Fourier excursions 0.25 and -0.24 $eÅ^{-3}$. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1c).

2,4,6-Trinitro-8,10-diacetyl-2,4,6,8,10-pentaazabicyclo[5.3.0]decane

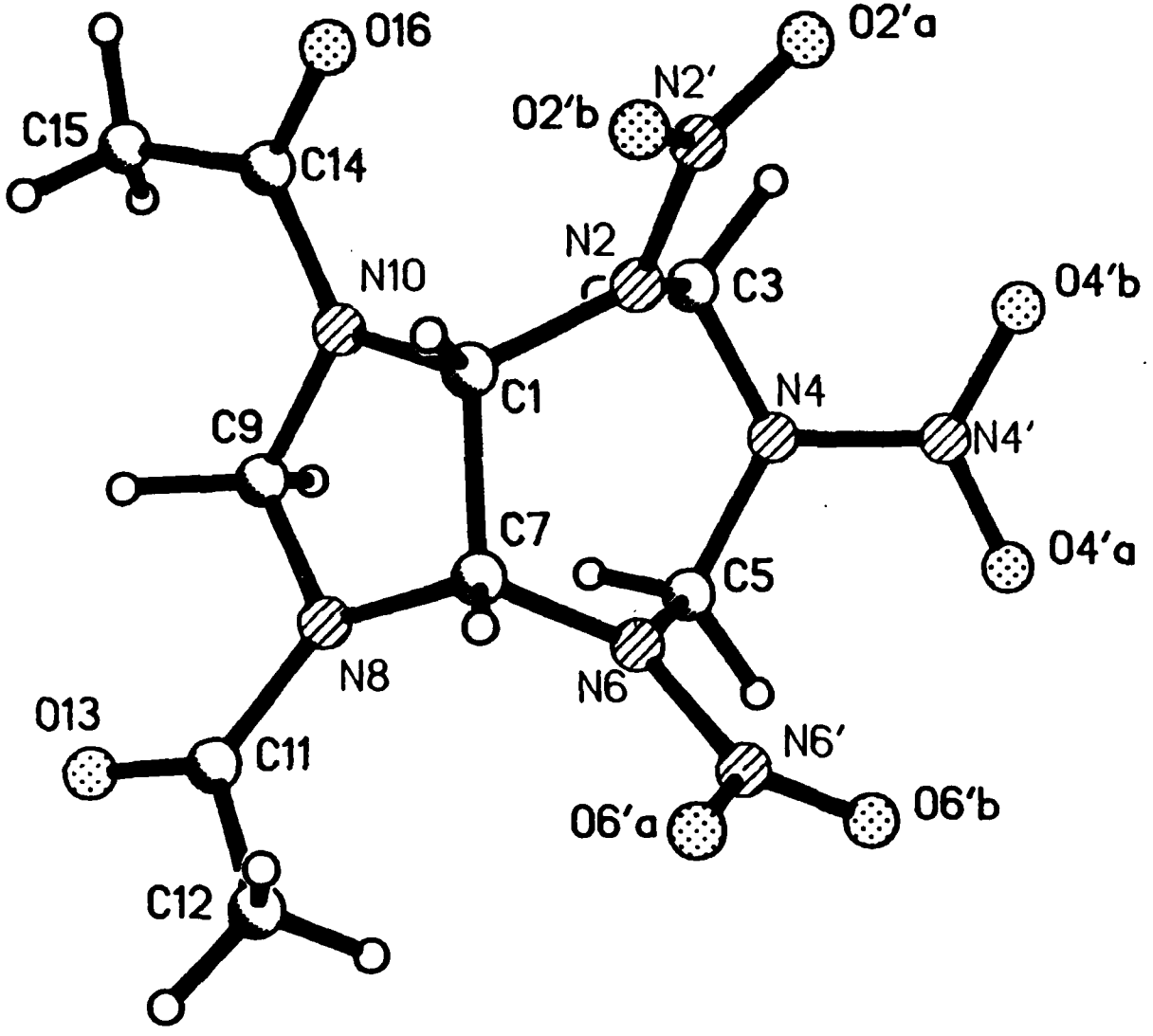


Table 1c. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 x10^3$)

	x	y	z	U(eq)
C(1)	1284(5)	1324(3)	5238(2)	26(1)
N(2)	13(4)	1173(2)	4532(2)	29(1)
C(3)	-1495(5)	607(3)	4779(3)	39(1)
N(4)	-1315(4)	-571(3)	4723(2)	38(1)
C(5)	-39(5)	-1096(3)	5260(3)	37(1)
N(6)	1614(4)	-717(2)	5045(2)	31(1)
C(7)	2319(4)	280(3)	5432(2)	26(1)
N(8)	2450(4)	266(2)	6452(2)	31(1)
C(9)	1212(5)	938(3)	6911(3)	36(1)
N(10)	640(4)	1636(2)	6147(2)	29(1)
N(2')	-56(4)	1943(3)	3808(2)	39(1)
O(2'A)	-1351(4)	2020(3)	3378(2)	58(1)
O(2'B)	1182(4)	2462(3)	3638(2)	52(1)
N(4')	-1880(4)	-1092(3)	3905(3)	46(1)
O(4'A)	-1674(5)	-2076(3)	3856(3)	65(1)
O(4'B)	-2627(4)	-536(3)	3333(2)	57(1)
N(6')	2568(5)	-1349(3)	4448(2)	39(1)
O(6'A)	3971(4)	-1054(3)	4293(2)	48(1)
O(6'B)	1946(4)	-2176(2)	4130(2)	58(1)
C(11)	3814(5)	-23(3)	6971(3)	33(1)
C(12)	5257(5)	-475(4)	6466(3)	49(1)
O(13)	3782(4)	91(2)	7824(2)	49(1)
C(14)	53(5)	2677(3)	6260(3)	30(1)
C(15)	-310(6)	3041(3)	7237(3)	43(1)
O(16)	-99(3)	3261(2)	5565(2)	38(1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2c. Bond lengths (Å)

C(1)-N(2)	1.452(5)	C(1)-C(7)	1.555(5)
C(1)-N(10)	1.444(4)	N(2)-C(3)	1.454(5)
N(2)-N(2')	1.395(4)	C(3)-N(4)	1.450(5)
N(4)-C(5)	1.440(5)	N(4)-N(4')	1.401(5)
C(5)-N(6)	1.456(5)	N(6)-C(7)	1.457(4)
N(6)-N(6')	1.385(4)	C(7)-N(8)	1.450(4)
N(8)-C(9)	1.455(5)	N(8)-C(11)	1.379(5)
C(9)-N(10)	1.455(5)	N(10)-C(14)	1.370(5)
N(2')-O(2'A)	1.222(5)	N(2')-O(2'B)	1.216(5)
N(4')-O(4'A)	1.217(5)	N(4')-O(4'B)	1.222(5)
C(11)-C(12)	1.484(5)	C(11)-O(13)	1.218(5)
C(14)-C(15)	1.485(5)	C(14)-O(16)	1.225(4)

Table 3c. Bond angles (°)

N(2)-C(1)-C(7)	113.9(3)	N(2)-C(1)-N(10)	113.0(3)
C(7)-C(1)-N(10)	104.8(3)	C(1)-N(2)-C(3)	119.8(3)
C(1)-N(2)-N(2')	116.8(3)	C(3)-N(2)-N(2')	117.7(3)
N(2)-C(3)-N(4)	112.0(3)	C(3)-N(4)-C(5)	119.1(3)
C(3)-N(4)-N(4')	117.7(3)	C(5)-N(4)-N(4')	118.1(3)
N(4)-C(5)-N(6)	114.5(3)	C(5)-N(6)-C(7)	123.5(3)
C(5)-N(6)-N(6')	118.0(3)	C(7)-N(6)-N(6')	118.5(3)
C(1)-C(7)-N(6)	114.0(3)	C(1)-C(7)-N(8)	103.1(3)
C(6)-C(7)-N(8)	113.3(3)	C(7)-N(8)-C(9)	112.9(3)
C(7)-N(8)-C(11)	126.5(3)	C(9)-N(8)-C(11)	117.6(3)
N(8)-C(9)-N(10)	102.7(3)	C(1)-N(10)-C(9)	113.2(3)
C(1)-N(10)-C(14)	118.5(3)	C(9)-N(10)-C(14)	124.8(3)
N(2)-N(2')-O(2'A)	117.1(3)	N(2)-N(2')-O(2'B)	117.7(3)
O(2'A)-N(2')-O(2'B)	125.2(3)	N(4)-N(4')-O(4'A)	116.9(3)
N(4)-N(4')-O(4'B)	117.4(3)	O(4'A)-N(4)-O(4'B)	125.6(4)
N(6)-N(6')-O(6'A)	118.1(3)	N(6)-N(6')-O(6'B)	117.2(3)
O(6'A)-N(6')O(6'B)	124.7(4)	N(8)-C(11)-C(12)	118.4(4)
N(8)-C(11)-O(13)	119.0(4)	C(12)-C(11)-O(13)	122.6(3)
N(10)-C(14)-C(15)	117.2(3)	N(10)-C(14)-O(16)	118.9(3)
C(15)-C(14)-O(16)	123.8(3)		

Abstract

2,4-Dinitro-6,8,-dipropionyl-2,4,6,8-tetraazabicyclo[3.3.0]octane, $C_{10}H_{16}N_6O_6$, M_r = 316.27, monoclinic, C2/c, a = 23.822(7), b = 6.163(2), c = 20.300(5) Å, β = 106.54(2)°, V = 2857.0(12) Å^3, Z = 8, D_x = 1.471 Mg m^{-3}, λ(Mo Kα) = 0.71069 Å, μ = 8.10 cm^{-1}, F(000) = 1328, T = 295 K, Final R = 0.075, wR = 0.064 for 2011 independent reflections. All four of the ring nitrogens are directed away from the cleft of the two rings. The two ring nitrogens involved in N-N bonding are pyramidal;the angles between the exocyclic N-N bonds and the C-N-C plane are 44.4 and 42.5°. The terminal atoms of one of the propionyl groups shows large thermal motion but no disorder can be discerned in the difference maps.

Experimental

A clear colorless 0.05 x 0.10 x 0.20 mm data crystal recrystallized from ethyl acetate was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within $18 \leq 2\theta \leq 27°$ used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.55 Å^{-1}, range of hkl: $-26 \leq h \leq 24$, $0 \leq k \leq 6$, $0 \leq \leq 20$. Standards 600, 332, 006, monitored every 60 reflections with random variation of 2.0 % over data collection, θ/2θ mode, scan width $[2\theta(K\alpha_1) - 0.6]$ to $[2\theta(K\alpha_2) + 0.6]°$, scan rate a function of count rate (8.0/min. minimum, 30.0°/min. maximum), 2497 reflections measured, 2011 unique, R_{int} = 0.04, 1307 observed with $Fo > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorbtion effects . Structure solved by direct methods. The least-squares refinement used the MicroVAX version of program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. 221 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model (coordinate shifts of C applied to attached H atoms), C-H = 0.96 Å, C-C-H = 109.5° (methyl H) , $U(H) = 1.1\ U_{eq}(C)$. (Δ/σ) max = 0.345, R = 0.075, wR = 0.064, S = 1.44. Final difference Fourier excursions 0.34 and -0.32 eÅ^{-3}. Atomic scattering factors from

International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1d).

2,4-Diacetyl-6-nitro-2,4,6-triaza-8-oxabicyclo[3.3.0]octane

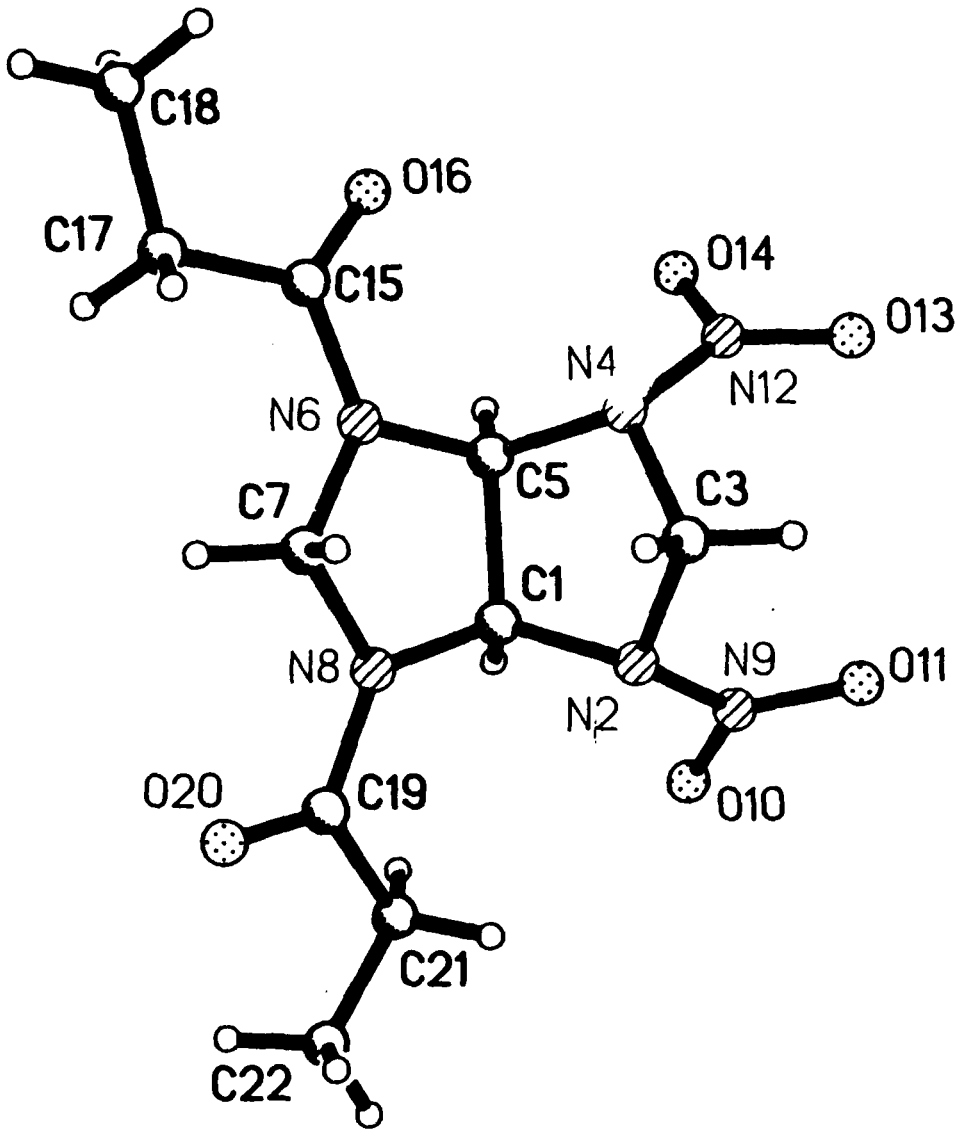


Table 1d. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2 x 10^2$)

	x	y	z	U(eq)
C(1)	0.5790(2)	0.0841(10)	0.3632(3)	3.4(2)
N(2)	0.5313(2)	0.2419(8)	0.3581(2)	3.2(2)
C(3)	0.5082(3)	0.1990(9)	0.4165(3)	3.5(2)
N(4)	0.5148(2)	-.0333(7)	0.4286(2)	3.4(2)
C(5)	0.5675(2)	-.1051(10)	0.4087(3)	3.3(2)
N(6)	0.6191(2)	-.1124(8)	0.4666(2)	3.8(2)
C(7)	0.6542(3)	0.0841(10)	0.4730(3)	4.4(3)
N(8)	0.6355(2)	0.1678(8)	0.4023(2)	4.0(2)
N(9)	0.4897(2)	0.2515(8)	0.2930(3)	4.1(2)
O(10)	0.5087(2)	0.2410(7)	0.2432(2)	5.2(2)
O(11)	0.4389(2)	0.2890(7)	0.2912(2)	5.5(2)
N(12)	0.4636(2)	-.1569(9)	0.4023(3)	4.1(2)
O(13)	0.4170(2)	-.0696(8)	0.3997(2)	5.8(2)
O(14)	0.4692(2)	-.3474(7)	0.3891(2)	5.2(2)
C(15)	0.6286(3)	-.2843(10)	0.5114(3)	4.1(2)
O(16)	0.5940(2)	-.4334(7)	0.5011(2)	5.3(2)
C(17)	0.6833(3)	-.2718(11)	0.5721(3)	4.6(3)
C(18)	0.6859(5)	-.4468(14)	0.6234(4)	8.5(4)
C(19)	0.6672(3)	0.3279(12)	0.3835(4)	5.9(3)
O(20)	0.7098(2)	0.4024(9)	0.4251(3)	8.9(2)
C(21)	0.6466(4)	0.3955(21)	0.3084(5)	10.5(5)
C(22)	0.6856(6)	0.5147(40)	0.2845(9)	24.5(12)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2d. Bond lengths (Å)

C(1)-N(2)	1.478(7)	C(1)-C(5)	1.560(7)
C(1)-N(8)	1.448(7)	N(2)-C(3)	1.465(7)
N(2)-N(9)	1.409(6)	C(3)-N(4)	1.454(6)
N(4)-C(5)	1.493(7)	N(4)-N(12)	1.407(6)
C(5)-N(6)	1.439(6)	N(6)-C(7)	1.456(7)
N(6)-C(15)	1.372(7)	C(7)-N(8)	1.470(7)
N(8)-C(19)	1.361(7)	N(9)-O(10)	1.223(6)
N(9)-O(11)	1.221(6)	N(12)-O(13)	1.222(6)
N(12)-O(14)	1.220(6)	C(15)-O(16)	1.212(7)
C(15)-C(17)	1.519(8)	C(17)-C(18)	1.488(8)
C(19)-O(20)	1.211(7)	C(19)-C(21)	1.521(9)
C(21)-C(22)	1.377(12)		

Table 3d. Bond angles (°)

C(5)-C(1)-N(2)	105.8(4)	N(8)-C(1)-N(2)	112.4(5)
N(8)-C(1)-C(5)	103.6(4)	C(3)-N(2)-C(1)	106.5(4)
N(9)-N(2)-C(1)	114.6(4)	N(9)-N(2)-C(3)	116.1(5)
N(4)-C(3)-N(2)	105.5(5)	C(5)-N(4)-C(3)	107.7(5)
N(12)-N(4)-C(3)	115.4(5)	N(12)-N(4)-C(5)	116.2(4)
N(4)-C(5)-C(1)	104.1(4)	N(6)-C(5)-C(1)	104.6(4)
N(6)-C(5)-N(4)	112.2(5)	C(7)-N(6)-C(5)	112.5(5)
C(15)-N(6)-C(5)	120.4(5)	C(15)-N(6)-C(7)	126.9(5)
N(8)-C(7)-N(6)	101.4(5)	C(7)-N(8)-C(1)	112.7(5)
C(19)-N(8)-C(1)	127.5(5)	C(19)-N(8)-C(7)	119.3(5)
O(10)-N(9)-N(2)	116.5(5)	O(11)-N(9)-N(2)	117.6(5)
O(11)-N(9)-O(10)	125.6(5)	O(13)-N(12)-N(4)	117.1(5)
O(14)-N(12)-N(4)	117.9(5)	O(14)-N(12)-O(13)	124.7(6)
O(16)-C(15)-N(6)	119.7(5)	C(17)-C(15)-N(6)	116.1(5)
C(17)-C(15)-O(16)	124.2(6)	C(18)-C(17)-C(15)	112.7(6)
O(20)-C(19)-N(8)	120.2(6)	C(21)-C(19)-N(8)	115.6(6)
C(21)-C(19)-O(20)	124.2(7)	C(22)-C(21)-C(19)	116.2(9)

Abstract

2,4-Diacetyl-6-nitro-2,4,6-triaza-8-oxabicyclo[3.3.0]octane, $C_8H_{12}N_4O_5$, M_r = 244.21, monoclinic, $P2_1/c$, a = 7.801(1), b = 19.885(3), c = 7.077(1) Å, β = 91.4(1)°, V = 1097.5(3) Å^3, Z = 4, D_x = 1.478 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 10.24 cm^{-1}, F(000) = 512, T = 295 K, Final R = 0.036, wR = 0.049 for 1718 independent reflections. The Acetyl substituted five-membered ring is planar (average absolute ring torsion is 5.1°). The nitramine ring is an envelope with C(7) being the out of plane atom. The nitramine group is pyramidal with a C-N to C-N-C angle of 42.6°. Both methyl groups have disordered hydrogens with occupancies of 60:40.

Experimental

A clear colorless 0.20 x 0.60 x 0.35 mm data crystal recrystallized from ethyl acetate was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within 30 ≤ 2θ ≤ 82° used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.55 Å^{-1}, range of hkl : -8 ≤ h ≤ 0, -22 ≤ k ≤ 19, -8 ≤ l ≤ 9. Standards 600, 080, 004, monitored every 60 reflections with random variation of 2.0 % over data collection, θ/2θ mode, scan width [2θ(Kα_1) - 1.0] to [2θ(Kα_2) + 1.0]°, scan rate a function of count rate (4.0°/min. minimum, 30.0°/min. maximum), 4679 reflections measured, 1874 unique, R_{int} = 0.015, 1718 observed with Fo>3σ(F_o). Data corrected for Lorentz and polarization, but not absorbtion effects. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. 193 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model , C-H = 0.96 Å, C-C-H = 109.5° , U(H) = 1.1 U_{eq}(C) . Methyl hydrogens disordered, restrained to tetrahedral angles allowing only torsions to vary, occupancy refined to 60:40 for both methyl groups, (Δ/σ) max = 0.112, R = 0.036, wR = 0.049, S = 2.20. Final difference Fourier excursions 0.23 and -0.16 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray

Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1e).

2,4-Dinitro-6,8,-dipropionyl-2,4,6,8-tetraazabicyclo[3.3.0]octane

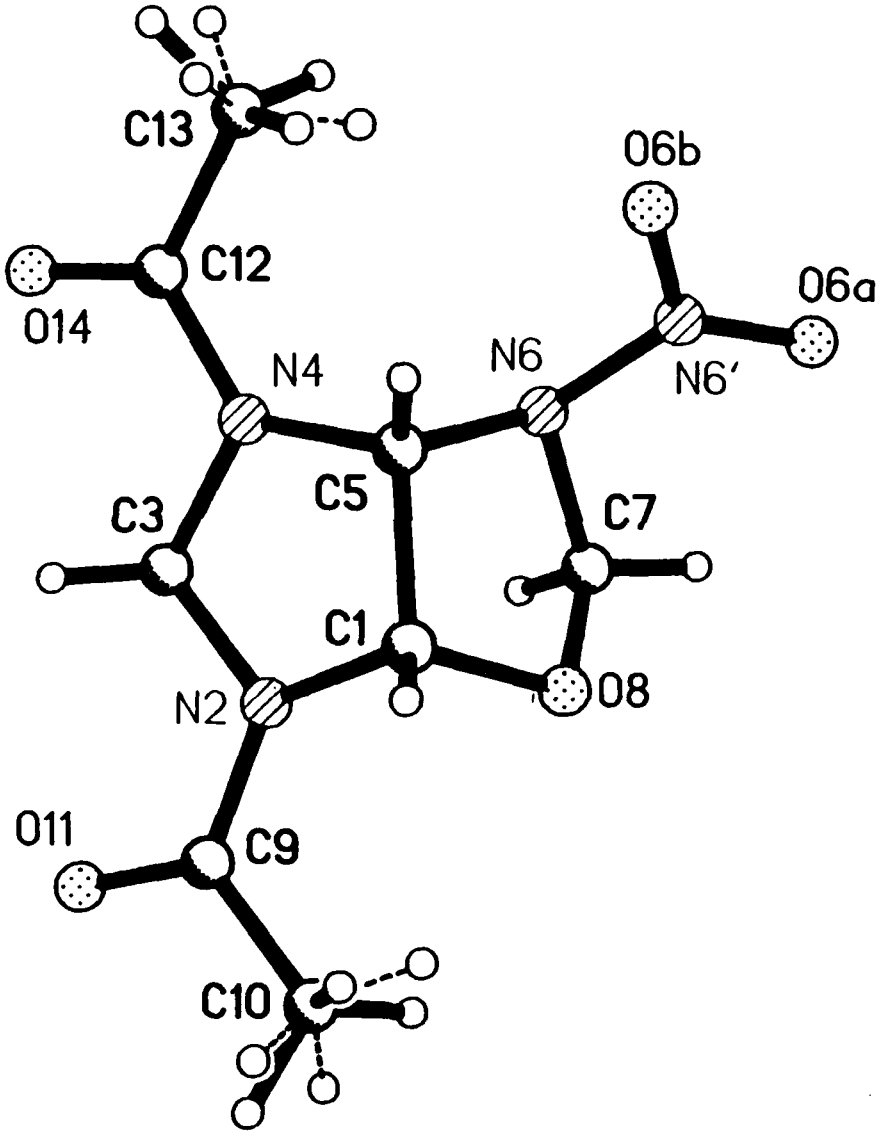


Table 1e. Atomic coordinates and equivalent isotropic displacement parameters ($A^2 x 10^2$)

	x	y	z	U(eq)
C(1)	0.3029(2)	0.3674(1)	0.4998(2)	3.7(1)
N(2)	0.2131(2)	0.3432(1)	0.3325(2)	3.8(1)
C(3)	0.0294(2)	0.3371(1)	0.3537(2)	4.6(1)
N(4)	0.0051(2)	0.3563(1)	0.5489(2)	4.1(1)
C(5)	0.1589(2)	0.3809(1)	0.6411(2)	3.7(1)
N(6)	0.1577(2)	0.4557(1)	0.6568(2)	4.3(1)
C(7)	0.2562(2)	0.4794(1)	0.4968(3)	4.9(1)
O(8)	0.3853(1)	0.4303(1)	0.4743(2)	4.8(1)
C(9)	0.2871(2)	0.3150(1)	0.1795(2)	4.4(1)
C(10)	0.4777(3)	0.3187(1)	0.1687(3)	6.8(1)
O(11)	0.1972(2)	0.2892(1)	0.0556(2)	5.9(1)
C(12)	-.1466(2)	0.3421(1)	0.6311(3)	5.0(1)
C(13)	-.1651(3)	0.3591(1)	0.8346(3)	7.1(1)
O(14)	-.2626(2)	0.3173(1)	0.5367(2)	7.3(1)
N(6')	0.2163(2)	0.4810(1)	0.8336(2)	6.1(1)
O(6A)	0.2789(3)	0.5367(1)	0.8340(3)	9.8(1)
O(6B)	0.1848(3)	0.4481(1)	0.9733(2)	8.9(1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2e. Bond lengths (A)

C(1)-N(2)	1.444 (2)	C(1)-C(5)	1.545 (2)
C(1)-O(8)	1.420 (2)	N(2)-C(3)	1.450 (2)
N(2)-C(9)	1.359 (2)	C(3)-N(4)	1.449 (2)
N(4)-C(5)	1.438 (2)	N(4)-C(12)	1.361 (2)
C(5)-N(6)	1.492 (2)	N(6)-C(7)	1.462 (2)
N(6)-N(6')	1.414 (2)	C(7)-O(8)	1.414 (2)
C(9)-C(10)	1.492 (3)	C(9)-O(11)	1.223 (2)
C(12)-C(13)	1.490 (3)	C(12)-O(14)	1.216 (2)
N(6')-O(6A)	1.211 (2)	N(6')-O(6B)	1.215 (2)

Table 3e. Bond angles (°)

C(5)-C(1)-N(2)	104.0(1)	O(8)-C(1)-N(2)	113.7(1)
O(8)-C(1)-C(5)	105.5(1)	C(3)-N(2)-C(1)	113.9(1)
C(9)-N(2)-C(1)	125.7(1)	C(9)-N(2)-C(3)	119.2(1)
N(4)-C(3)-N(2)	103.2(1)	C(5)-N(4)-C(3)	113.3(1)
C(12)-N(4)-C(3)	119.1(1)	C(12)-N(4)-C(5)	126.9(1)
N(4)-C(5)-C(1)	105.0(1)	N(6)-C(5)-C(1)	103.1(1)
N(6)-C(5)-N(4)	111.5(1)	C(7)-N(6)-C(5)	105.1(1)
N(6')-N(6)-C(5)	114.7(1)	N(6')-N(6)-C(7)	113.9(1)
O(8)-C(7)-N(6)	104.7(1)	C(7)-O(8)-C(1)	105.6(1)
C(10)-C(9)-N(2)	117.5(1)	O(11)-C(9)-N(2)	119.8(2)
O(11)-C(9)-C(10)	122.6(2)	C(13)-C(12)-N(4)	118.2(2)
O(14)-C(12)-N(4)	119.5(2)	O(14)-C(12)-C(13)	122.3(2)
O(6A)-N(6')-N(6)	116.7(2)	O(6B)-N(6')-N(6)	117.5(1)
O(6B)-N(6')-O(6A)	125.4(2)		

Abstract

2,4,6,8-Tetrabenzyl-2,4,6,8-tetraazabicyclo[3.3.0]octane, $C_{32}H_{34}N_4$, M_r = 474.65, triclinic, $P\bar{1}$, a = 10.952(2), b = 12.069(2), c = 22.038(4) Å, α = 75.44(1), β = 83.03(2), γ = 77.66(10)°, V = 2746.8(4) Å^3, Z = 4 (two molecules per asymmetric unit), D_x = 1.148 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 4.92 cm^{-1}, F(000) = 1015, T = 295 K, Final R = 0.073, wR = 0.065 for 4435 independent reflections.

Experimental

A clear colorless 0.10 x 0.10 x 0.41 mm data crystal recrystallized from methanol was provided by A. Nielsen of NWC, China Lake. Automated Nicolet R3m diffractometer with incident beam monochromator. 15 centered reflections within 41 ≤ 2θ ≤ 63° used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.55 Å^{-1}, range of hkl : -11 ≤ h ≤ 11, 0 ≤ k ≤ 13, -23 ≤ l ≤ 24. Standards 0 0 10, 401, 166, monitored every 60 reflections with random variation of 2.1 % over data collection, $\theta/2\theta$ mode, scan width [2θ(Kα_1) - 1.0] to [2θ(Kα_2) + 1.0]°, scan rate a function of count rate (8.0°/min. minimum, 30.0°/min. maximum), 8411 reflections measured, 7544 unique, R_{int} = 0.07, 4435 observed with Fo>3σ(F_o). Data corrected for Lorentz and polarization, but not absorbtion effects . Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.0004. Secondary extinction parameter p = 0.0008(2) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin 2\theta]^{0.25}$. 650 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, benzene group atoms constrained to move as rigid groups, H atoms included using riding model (coordinate shifts of C applied to attached H atoms), C-H = 0.96 Å, C-C-H = 109.5° (methyl H) , $U(H) = 1.1U_{eq}(C)$. (Δ/σ) max = 0.18, R = 0.073, wR = 0.065, S = 1.352. Final difference Fourier excursions 0.23 and -0.18 eÅ^3. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, and 2 , atom coordinates, bond distances and angles, follows that shown in Fig.(1f).

2,4,6,8-Tetrabenzyl-2,4,6,8-tetraazabicyclo[3.3.0]octane

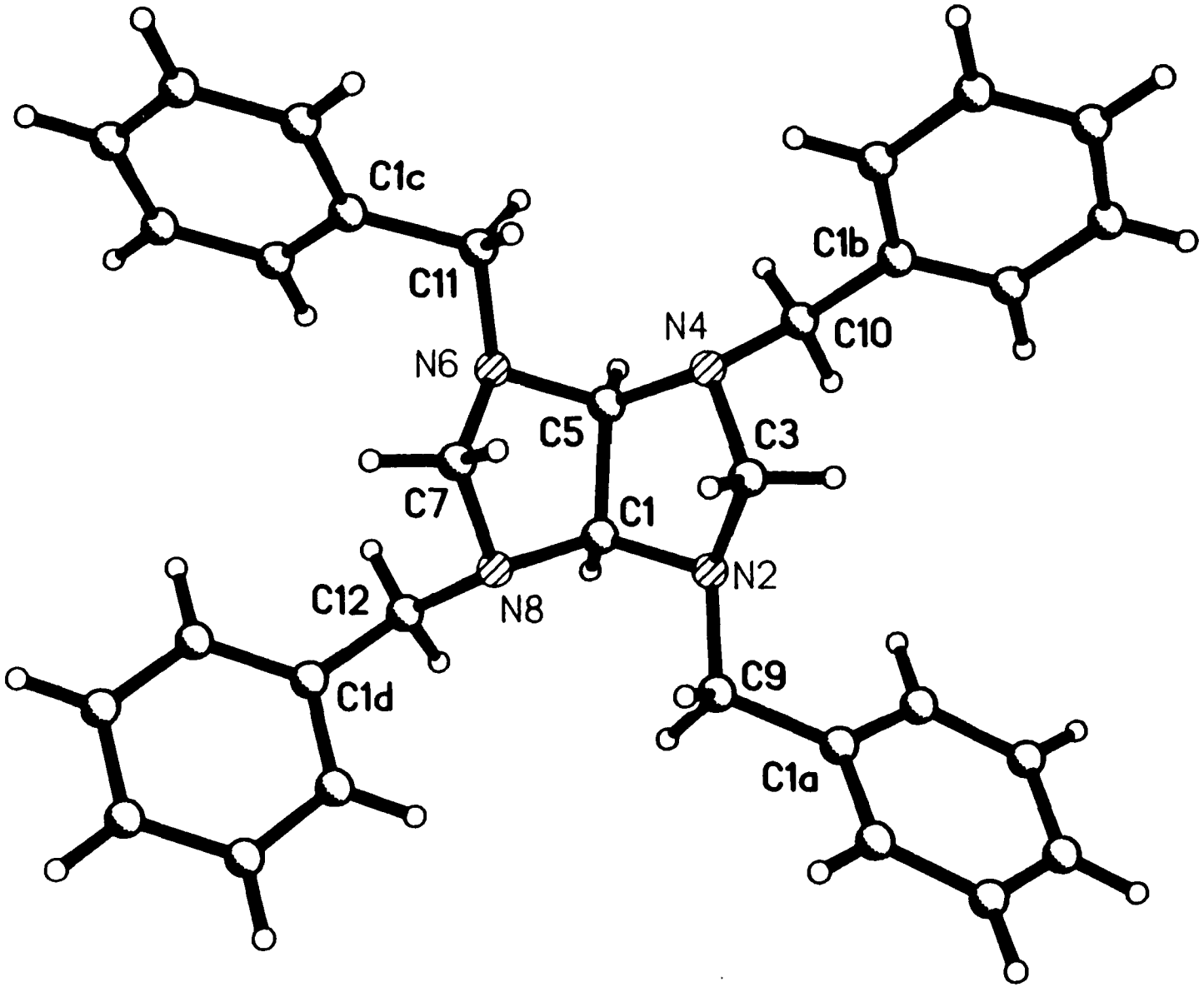


Table 1f. Atom coordinates ($x10^4$) and thermal parameters ($Å^2 \times 10^3$)

atom	x	y	z	U
C(1)	305(3)	8924(3)	2899(2)	54(1)*
N(2)	1016(3)	8770(2)	2317(1)	61(1)*
C(3)	294(3)	9569(3)	1812(2)	68(2)*
N(4)	-1010(3)	9533(2)	2017(1)	60(1)*
C(5)	-1059(3)	9259(3)	2714(2)	58(2)*
N(6)	-1567(2)	10241(2)	3001(1)	57(1)*
C(7)	-532(3)	10832(3)	2975(2)	61(2)*
N(8)	538(2)	9894(2)	3144(1)	53(1)*
C(9)	2328(3)	8855(3)	2283(2)	74(2)*
C(2a)	4225(4)	8620(4)	1536(2)	86(2)*
C(3a)	5041(4)	8029(5)	1137(2)	110(3)*
C(4a)	4753(5)	7071(5)	1015(3)	118(3)*
C(5a)	3664(5)	6716(4)	1265(2)	106(3)*
C(6a)	2850(4)	7296(3)	1668(2)	83(2)*
C(1a)	3147(3)	8263(3)	1814(2)	64(2)*
C(10)	-1465(4)	8658(3)	1785(2)	70(2)*
C(2b)	-1185(4)	8366(4)	697(2)	103(2)*
C(3b)	-1460(4)	8688(4)	72(2)	122(3)*
C(4b)	-2220(4)	9710(4)	-154(2)	96(2)*
C(5b)	-2750(4)	10384(4)	241(2)	100(2)*
C(6b)	-2485(4)	10057(3)	867(2)	87(2)*
C(1b)	-1698(3)	9047(3)	1101(2)	58(2)*
C(11)	-2731(3)	10972(3)	2781(2)	75(2)*
C(2c)	-4048(4)	12805(4)	3029(3)	115(3)*
C(3c)	-4647(4)	13435(4)	3459(3)	165(4)*
C(4c)	-4564(5)	13039(6)	4074(3)	179(4)*
C(5c)	-3890(4)	11934(5)	4303(3)	135(3)*
C(6c)	-3288(4)	11276(4)	3879(2)	93(2)*
C(1c)	-3359(3)	11697(3)	3246(2)	78(2)*
C(12)	644(3)	9568(3)	3826(1)	63(2)*
C(2d)	587(4)	11139(3)	4412(2)	77(2)*
C(3d)	1163(5)	11916(4)	4591(2)	92(2)*
C(4d)	2375(5)	11980(4)	4389(2)	92(2)*
C(5d)	3013(4)	11294(4)	4008(2)	98(2)*
C(6d)	2450(4)	10520(4)	3833(2)	82(2)*
C(1d)	1233(4)	10428(3)	4032(2)	62(2)*
C(1')	-558(3)	16155(3)	7102(2)	57(1)*
N(2')	-676(2)	15291(2)	6765(1)	58(1)*
C(3')	209(3)	14238(3)	7031(2)	65(2)*
N(4')	1311(3)	14646(2)	7134(1)	55(1)*
C(5')	813(3)	15816(3)	7263(2)	57(2)*
N(6')	741(3)	15857(2)	7916(1)	61(1)*
C(7')	-452(3)	15551(3)	8185(2)	70(2)*
N(8')	-1328(3)	16108(2)	7702(1)	58(1)*
C(9')	-1946(3)	15125(3)	6755(2)	68(2)*
C(6a')	-1352(4)	14594(3)	5709(2)	75(2)*
C(5a')	-1553(4)	14072(4)	5249(2)	85(2)*
C(4a')	-2503(5)	13477(4)	5329(2)	89(2)*
C(3a')	-3244(4)	13388(4)	5869(2)	92(2)*
C(2a')	-3033(4)	13883(3)	6338(2)	75(2)*
C(1a')	-2096(3)	14517(3)	6256(2)	58(2)*
C(10')	2221(3)	14710(3)	6582(2)	68(2)*
C(6b')	3726(3)	12853(3)	6974(2)	74(2)*
C(5b')	4408(3)	11771(4)	6921(2)	82(2)*
C(4b')	4298(4)	11354(3)	6411(2)	85(2)*
C(3b')	3530(4)	11999(3)	5960(2)	91(2)*
C(2b')	2861(4)	13078(3)	6017(2)	80(2)*
C(1b')	2942(3)	13518(3)	6525(2)	58(2)*
C(11')	1815(3)	15247(3)	8269(2)	77(2)*

C(6c')	1493(4)	16728(4)	8918(2)	89(2)*
C(5c')	1459(5)	17032(5)	9481(3)	114(3)*
C(4c')	1687(5)	16202(7)	10014(3)	130(4)*
C(3c')	1961(5)	15068(6)	9995(2)	135(3)*
C(2c')	1995(4)	14756(4)	9431(2)	96(2)*
C(1c')	1758(3)	15566(4)	8888(2)	68(2)*
C(12')	-1990(3)	17270(3)	7778(2)	75(2)*
C(6d')	-3869(4)	16508(4)	8339(2)	94(2)*
C(5d')	-4844(4)	16462(4)	8797(2)	112(3)*
C(4d')	-4946(5)	17098(4)	9234(3)	107(3)*
C(3d')	-4093(5)	17773(4)	9214(2)	112(3)*
C(2d')	-3122(4)	17823(3)	8750(2)	93(2)*
C(1d')	-3009(4)	17198(3)	8299(2)	64(2)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U tensor

Table 2f. Bond Lengths (Å) and bond angles (deg.)

C(1)-N(2)	1.450(4)	C(1)-C(5)	1.538(5)
C(1)-N(8)	1.485(5)	N(2)-C(3)	1.463(4)
N(2)-C(9)	1.453(5)	C(3)-N(4)	1.450(4)
N(4)-C(5)	1.485(4)	N(4)-C(10)	1.478(5)
C(5)-N(6)	1.456(4)	N(6)-C(7)	1.452(5)
N(6)-C(11)	1.446(4)	C(7)-N(8)	1.456(4)
N(8)-C(12)	1.468(4)	C(9)-C(1a)	1.501(5)
C(2a)-C(3a)	1.389(7)	C(2a)-C(1a)	1.366(5)
C(3a)-C(4a)	1.357(10)	C(4a)-C(5a)	1.360(8)
C(5a)-C(6a)	1.389(6)	C(6a)-C(1a)	1.395(6)
C(10)-C(1b)	1.497(5)	C(2b)-C(3b)	1.386(6)
C(2b)-C(1b)	1.353(6)	C(3b)-C(4b)	1.348(6)
C(4b)-C(5b)	1.337(6)	C(5b)-C(6b)	1.385(6)
C(6b)-C(1b)	1.356(5)	C(11)-C(1c)	1.518(6)
C(2c)-C(3c)	1.379(8)	C(2c)-C(1c)	1.386(5)
C(3c)-C(4c)	1.328(10)	C(4c)-C(5c)	1.382(8)
C(5c)-C(6c)	1.391(8)	C(6c)-C(1c)	1.367(6)
C(12)-C(1d)	1.515(6)	C(2d)-C(3d)	1.390(7)
C(2d)-C(1d)	1.376(5)	C(3d)-C(4d)	1.359(8)
C(4d)-C(5d)	1.355(7)	C(5d)-C(6d)	1.374(7)
C(6d)-C(1d)	1.371(6)	C(1')-N(2')	1.458(5)
C(1')-C(5')	1.530(5)	C(1')-N(8')	1.475(4)
N(2')-C(3')	1.462(4)	N(2')-C(9')	1.450(5)
C(3')-N(4')	1.459(5)	N(4')-C(5')	1.486(4)
N(4')-C(10')	1.474(4)	C(5')-N(6')	1.444(5)
N(6')-C(7')	1.450(5)	N(6')-C(11')	1.447(4)
C(7')-N(8')	1.456(4)	N(8')-C(12')	1.474(4)
C(9')-C(1a')	1.508(6)	C(6a')-C(5a')	1.381(7)
C(6a')-C(1a')	1.366(5)	C(5a')-C(4a')	1.357(7)
C(4a')-C(3a')	1.350(7)	C(3a')-C(2a')	1.379(7)
C(2a')-C(1a')	1.377(6)	C(10')-C(1b')	1.513(5)
C(6b')-C(5b')	1.384(6)	C(6b')-C(1b')	1.369(5)
C(5b')-C(4b')	1.370(7)	C(4b')-C(3b')	1.355(5)
C(3b')-C(2b')	1.378(5)	C(2b')-C(1b')	1.375(6)
C(11')-C(1c')	1.499(6)	C(6c')-C(5c')	1.372(8)
C(6c')-C(1c')	1.387(7)	C(5c')-C(4c')	1.348(8)
C(4c')-C(3c')	1.348(11)	C(3c')-C(2c')	1.378(8)
C(2c')-C(1c')	1.354(6)	C(12')-C(1d')	1.501(5)
C(6d')-C(5d')	1.378(6)	C(6d')-C(1d')	1.367(7)
C(5d')-C(4d')	1.356(8)	C(4d')-C(3d')	1.355(8)
C(3d')-C(2d')	1.383(7)	C(2d')-C(1d')	1.371(6)

TABLE 2f. Bond angles (deg.)

N(2)-C(1)-C(5)	103.1(3)	N(2)-C(1)-N(8)	115.2(3)
C(5)-C(1)-N(8)	106.6(2)	C(1)-N(2)-C(3)	105.9(2)
C(1)-N(2)-C(9)	115.2(3)	C(3)-N(2)-C(9)	115.3(3)
N(2)-C(3)-N(4)	105.6(2)	C(3)-N(4)-C(5)	105.2(3)
C(3)-N(4)-C(10)	111.9(3)	C(5)-N(4)-C(10)	111.9(2)
C(1)-C(5)-N(4)	106.9(2)	C(1)-C(5)-N(6)	102.5(3)
N(4)-C(5)-N(6)	115.5(2)	C(5)-N(6)-C(7)	105.6(2)
C(5)-N(6)-C(11)	116.9(3)	C(7)-N(6)-C(11)	116.1(3)
N(6)-C(7)-N(8)	104.5(2)	C(1)-N(8)-C(7)	104.8(3)
C(1)-N(8)-C(12)	112.4(2)	C(7)-N(8)-C(12)	111.0(2)
N(2)-C(9)-C(1a)	114.1(3)	C(3a)-C(2a)-C(1a)	122.2(5)
C(2a)-C(3a)-C(4a)	119.0(5)	C(3a)-C(4a)-C(5a)	120.4(5)
C(4a)-C(5a)-C(6a)	120.8(5)	C(5a)-C(6a)-C(1a)	119.6(4)
C(9)-C(1a)-C(2a)	121.5(4)	C(9)-C(1a)-C(6a)	120.7(3)
C(2a)-C(1a)-C(6a)	117.8(3)	N(4)-C(10)-C(1b)	112.8(3)
C(3b)-C(2b)-C(1b)	121.3(4)	C(2b)-C(3b)-C(4b)	120.5(4)
C(3b)-C(4b)-C(5b)	118.9(4)	C(4b)-C(5b)-C(6b)	120.6(4)
C(5b)-C(6b)-C(1b)	121.5(4)	C(10)-C(1b)-C(6b)	121.0(3)
C(10)-C(1b)-C(6b)	121.7(3)	C(2b)-C(1b)-C(6b)	117.2(3)
N(6)-C(11)-C(1c)	111.0(3)	C(3c)-C(2c)-C(1c)	119.1(5)
C(2c)-C(3c)-C(4c)	122.7(5)	C(3c)-C(4c)-C(5c)	119.3(6)
C(4c)-C(5c)-C(6c)	119.0(5)	C(5c)-C(6c)-C(1c)	121.4(4)
C(11)-C(1c)-C(2c)	119.9(4)	C(11)-C(1c)-C(6c)	121.7(3)
C(2c)-C(1c)-C(6c)	118.4(4)	N(8)-C(12)-C(1d)	110.9(3)
C(3d)-C(2d)-C(1d)	120.6(4)	C(2d)-C(3d)-C(4d)	119.9(4)
C(3d)-C(4d)-C(5d)	119.9(5)	C(4d)-C(5d)-C(6d)	120.5(4)
C(5d)-C(6d)-C(1d)	121.1(4)	C(12)-C(1d)-C(2d)	122.4(4)
C(12)-C(1d)-C(6d)	119.5(3)	C(2d)-C(1d)-C(6d)	118.1(4)
N(2')-C(1')-C(5')	103.3(3)	N(2')-C(1')-N(8')	114.9(3)
C(5')-C(1')-N(8')	106.8(3)	C(1')-N(2')-C(3')	105.5(3)
C(1')-N(2')-C(9')	114.8(3)	C(3')-N(2')-C(9')	114.9(3)
N(2')-C(3')-N(4')	105.3(3)	C(3')-N(4')-C(5')	104.7(2)
C(3')-N(4')-C(10')	111.6(3)	C(5')-N(4')-C(10')	111.4(2)
C(1')-C(5')-N(4')	107.1(3)	C(1')-C(5')-N(6')	103.4(3)
N(4')-C(5')-N(6')	115.0(2)	C(5')-N(6')-C(7')	105.8(3)
C(5')-N(6')-C(11')	117.3(3)	C(7')-N(6')-C(11')	115.7(2)
N(6')-C(7')-N(8')	105.4(2)	C(1')-N(8')-C(7')	104.9(2)
C(1')-N(8')-C(12')	112.6(2)	C(7')-N(8')-C(12')	111.8(3)
N(2')-C(9')-C(1a')	113.4(3)	C(5a')-C(6a')-C(1a')	121.0(4)
C(6a')-C(5a')-C(4a')	120.6(4)	C(5a')-C(4a')-C(3a')	119.2(5)
C(4a')-C(3a')-C(2a')	120.7(5)	C(3a')-C(2a')-C(1a')	120.8(4)
C(9')-C(1a')-C(6a')	122.5(4)	C(9')-C(1a')-C(2a')	119.9(3)
C(6a')-C(1a')-C(2a')	117.6(4)	N(4')-C(10')-C(1b')	112.2(2)
C(5b')- C(6b')-C(1b')	121.2(4)	b')-C(5b')-C(4b')	119.6(3)
C(5b')-C(4b')-C(3b')	120.1(4)	C(4b')-C(3b')-C(2b')	119.7(4)
C(3b')-C(2b')-C(1b')	121.6(3)	C(10')-C(1b')-C(6b')	120.7(4)
C(10')-C(1b')-C(2b')	121.6(3)	C(6b')-C(1b')-C(2b')	117.7(3)
N(6')-C(11')-C(1c')	112.3(3)	C(5c')-C(6c')-C(1c')	120.7(4)
C(6c')-C(5c')-C(4c')	120.3(6)	C(5c')-C(4c')-C(3c')	120.0(6)
C(4c')-C(3c')-C(2c')	119.9(5)	C(3c')-C(2c')-C(1c')	121.6(5)
C(11')-C(1c')-C(6c')	120.2(3)	C(11')-C(1c')-C(2c')	122.5(4)
C(6c')-C(1c')-C(2c')	117.3(4)	N(8')-C(12')-C(1d')	112.2(3)
C(5d')-C(6d')-C(1d')	122.1(5)	C(6d')-C(5d')-C(4d')	119.5(5)
C(5d')-C(4d')-C(3d')	119.8(5)	C(4d')-C(3d')-C(2d')	120.5(5)
C(3d')-C(2d')-C(1d')	120.7(5)	C(12')-C(1d')-C(6d')	120.9(4)
C(12')-C(1d')-C(2d')	121.7(4)	C(6d')-C(1d')-C(2d')	117.3(4)

Abstract

2,5,7,9-Tetranitro-8-acetoxy-2,5,7,9-tetraazabicyclo[4.3.0]nonane, $C_7H_{10}N_8O_{10}$, M_r = 366.21, monoclinic , $P2_1/a$, a = 11.206(2), b = 10.757(2), c = 23.026(4) Å, β = 93.7(1)°, V = 2769.8(7) Å^3, Z = 8, D_x = 1.756 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 14.0 cm^{-1}, F(000) = 1504, T = 205 K, Final R = 0.062, wR = 0.070 for 1979 independent reflections. The two molecules in the asymmetric unit have essentially the same conformation.The five membered ring is an envelope and the six membered ring is non-planar. Each of these two rings contains a planar and a pyramidal N atom with the simalarly configured N atoms at opposing sides of the ring.

Experimental

A clear colorless 0.35 x 0.12 x 0.04 mm data crystal was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet R3m diffractometer with incident beam monochromator. 20 centered reflections within $17 \leq 2\theta \leq 70°$ used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.50 Å^{-1}, range of hkl : $-7 \leq h \leq 0$, $-10 \leq k \leq 0$, $-22 \leq l \leq 22$. Standards 200, 020, 0 0 10, monitored every 60 reflections with random variation of 2.0 % over data collection, $\theta/2\theta$ mode, scan width [$2\theta(K\alpha_1)$ - 1.0] to [$2\theta(K\alpha_2)$ + 1.0]°, scan rate a function of count rate (8.0°/min. minimum, 30.0°/min. maximum), 3433 reflections measured, 2436 unique, R_{int} = 0.032, 1979 observed with $Fo > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorbtion effects . Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.0004. 459 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model (coordinate shifts of C applied to attached H atoms), C-H = 0.96 Å, C-C-H = 109.5° (methyl H) , $U(H) = 1.1\ U_{eq}(C)$. (Δ/σ) max = 0.32, R = 0.062, wR = 0.070, S = 2.41. Final difference Fourier excursions 0.69 and -0.46 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1g).

2,5,7,9-Tetranitro-8-acetoxy-2,5,7,9-tetraazabicyclo[4.3.0]nonane

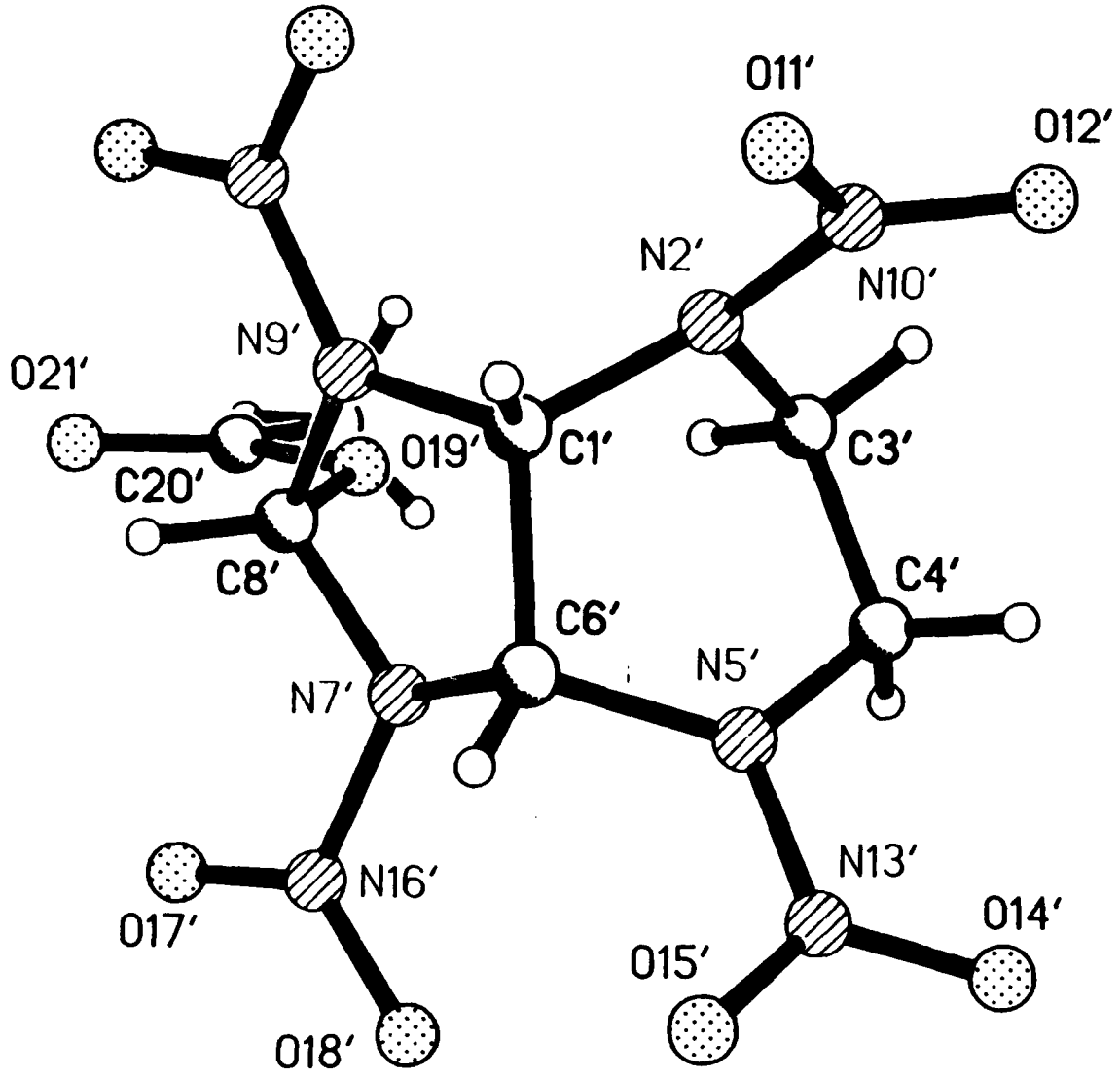


Table 1g. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2 x 10^2$)

	x	y	z	U(eq)
C(1)	0.9598(7)	-.1272(6)	0.3635(2)	2.7(3)
N(2)	0.0838(6)	-.1603(4)	0.3708(2)	3.1(2)
C(3)	0.1803(6)	-.0708(6)	0.3654(3)	3.4(3)
C(4)	0.1282(6)	0.0434(6)	0.3340(3)	3.7(3)
N(5)	0.0394(5)	0.0082(4)	0.2877(2)	3.1(2)
C(6)	0.9334(6)	-.0558(6)	0.3064(3)	2.9(3)
N(7)	0.8429(5)	0.0291(5)	0.3235(2)	3.2(2)
C(8)	0.8466(6)	0.0605(6)	0.3836(2)	3.1(3)
N(9)	0.9249(5)	-.0381(4)	0.4070(2)	2.8(2)
N(10)	0.1143(8)	-.2782(6)	0.3888(2)	3.7(3)
O(11)	0.2214(6)	-.2961(5)	0.4007(2)	5.1(2)
O(12)	0.0352(5)	-.3560(5)	0.3917(2)	4.8(2)
N(13)	0.0815(8)	-.0246(6)	0.2344(2)	4.6(3)
O(14)	0.1792(6)	0.0168(6)	0.2237(2)	6.9(3)
O(15)	0.0136(5)	-.0866(5)	0.2017(2)	5.7(2)
N(16)	0.7754(5)	0.0946(5)	0.2839(2)	1.0(2)
O(17)	0.7046(4)	0.1662(4)	0.3025(2)	4.6(2)
O(18)	0.7886(4)	0.0740(4)	0.2332(2)	4.6(2)
O(19)	0.9059(4)	0.1752(4)	0.3945(2)	3.3(2)
C(20)	0.8443(10)	0.2654(6)	0.4250(3)	4.5(4)
O(21)	0.7468(7)	0.2526(5)	0.4382(3)	8.0(3)
C(22)	0.9271(7)	0.3741(6)	0.4355(3)	5.4(3)
N(23)	0.9050(5)	-.0801(6)	0.4628(2)	3.3(2)
O(24)	0.9513(4)	-.1804(4)	0.4755(2)	4.0(2)
O(25)	0.8517(4)	-.0103(4)	0.4932(2)	4.2(2)
C(1')	0.5189(6)	0.0805(6)	0.8133(3)	3.3(3)
N(2')	0.5001(6)	-.0387(6)	0.7857(2)	5.1(3)
C(3')	0.4818(9)	-.1417(6)	0.8260(4)	6.2(4)
C(4')	0.5870(9)	-.1650(7)	0.8641(3)	7.3(4)
N(5')	0.6435(7)	-.0493(5)	0.8800(3)	5.6(3)
C(6')	0.5861(7)	0.0707(6)	0.8737(3)	3.7(3)
N(7')	0.4914(6)	0.0911(5)	0.9132(2)	4.0(2)
C(8')	0.3784(7)	0.1331(6)	0.8871(2)	3.5(3)
N(9')	0.4071(5)	0.1359(5)	0.8272(2)	3.4(2)
N(10')	0.5608(6)	-.0653(9)	0.7362(3)	6.6(3)
O(11')	0.5900(5)	0.0191(8)	0.7086(2)	8.1(3)
O(12')	0.5746(6)	-.1783(7)	0.7259(2)	8.7(3)
N(13')	0.7592(9)	-.0507(7)	0.9014(3)	6.3(4)
O(14')	0.8058(7)	-.1527(6)	0.9068(3)	9.7(3)
O(15')	0.8133(6)	0.0467(7)	0.9113(2)	7.4(3)
N(16')	0.5196(9)	0.1292(6)	0.9700(3)	5.5(3)
O(17')	0.4399(6)	0.1802(5)	0.9947(2)	6.6(3)
O(18')	0.6190(6)	0.0990(5)	0.9899(2)	6.6(3)
O(19')	0.2908(5)	0.0417(3)	0.8961(2)	3.3(2)
C(20')	0.1854(8)	0.0755(7)	0.9190(3)	4.0(3)
O(21')	0.1598(5)	0.1811(5)	0.9253(2)	6.9(2)
C(22')	0.1152(5)	-.0325(7)	0.9329(4)	5.8(3)
N(23')	0.3305(6)	0.1834(5)	0.7855(2)	3.7(3)
O(24')	0.2358(5)	0.2257(4)	0.8013(2)	4.9(2)
O(25')	0.3589(4)	0.1795(4)	0.7352(2)	4.8(2)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2g. Bond lengths (Å)

C(1)-N(2)	1.434(8)	C(1)-C(6)	1.536(8)
C(1)-N(9)	1.459(7)	N(2)-C(3)	1.459(7)
N(2)-N(10)	1.372(7)	C(3)-C(4)	1.523(9)
C(4)-N(5)	1.460(8)	N(5)-C(6)	1.462(7)
N(5)-N(13)	1.388(7)	C(6)-N(7)	1.439(7)
N(7)-C(8)	1.423(7)	N(7)-N(16)	1.346(7)
C(8)-N(9)	1.457(7)	C(8)-O(19)	1.416(7)
N(9)-N(23)	1.393(6)	N(10)-O(11)	1.228(7)
N(10)-O(12)	1.224(7)	N(13)-O(14)	1.222(7)
N(13)-O(15)	1.232(7)	N(16)-O(17)	1.204(6)
N(16)-O(18)	1.206(6)	O(19)-C(20)	1.406(8)
C(20)-O(21)	1.160(8)	C(20)-C(22)	1.503(11)
N(23)-O(24)	1.225(6)	N(23)-O(25)	1.211(6)
C(1')-N(2')	1.440(8)	C(1')-C(6')	1.543(8)
C(1')-N(9')	1.442(8)	N(2')-C(3')	1.468(9)
N(2')-N(10')	1.395(8)	C(3')-C(4')	1.445(11)
C(4')-N(5')	1.433(9)	N(5')-C(6')	1.445(9)
N(5')-N(13')	1.357(9)	C(6')-N(7')	1.457(8)
N(7')-C(8')	1.439(8)	N(7')-N(16')	1.389(8)
C(8')-N(9')	1.437(7)	C(8')-O(19')	1.415(7)
N(9')-N(23')	1.346(7)	N(10')-O(11'	1.168(8)
N(10')-O(12'	1.251(8)	N(13')-O(14'	1.218(8)
N(13')-O(15'	1.225(8)	N(16')-O(17'	1.220(8)
N(16')-O(18'	1.221(8)	O(19')-C(20'	1.372(8)
C(20')-O(21'	1.184(8)	C(20')-C(22'	1.451(9)
N(23')-O(24'	1.231(6)	N(23')-O(25'	1.221(6)

Table 3g. Bond angles (°)

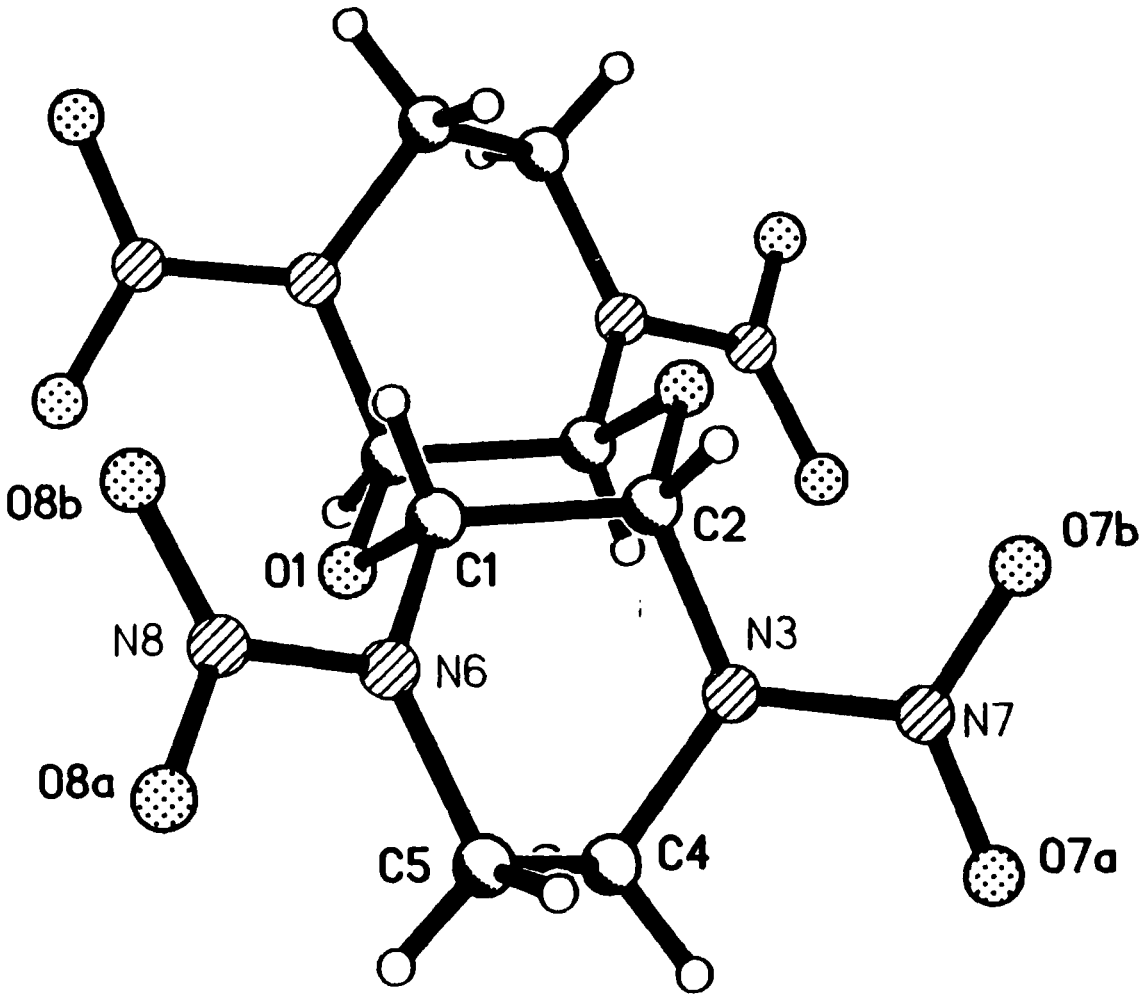
C(6)-C(1)-N(2)	110.9(5)	N(9)-C(1)-N(2)	112.5(5)
N(9)-C(1)-C(6)	102.3(5)	C(3)-N(2)-C(1)	122.9(5)
N(10)-N(2)-C(1)	119.1(6)	N(10)-N(2)-C(3)	117.7(7)
C(4)-C(3)-N(2)	108.0(5)	N(5)-C(4)-C(3)	111.1(5)
C(6)-N(5)-C(4)	115.9(5)	N(13)-N(5)-C(4)	117.1(6)
N(13)-N(5)-C(6)	118.0(5)	N(5)-C(6)-C(1)	111.8(5)
N(7)-C(6)-C(1)	100.5(5)	N(7)-C(6)-N(5)	112.5(5)
C(8)-N(7)-C(6)	116.2(5)	N(16)-N(7)-C(6)	121.4(5)
N(16)-N(7)-C(8)	121.0(5)	N(9)-C(8)-N(7)	99.5(5)
O(19)-C(8)-N(7)	111.2(5)	O(19)-C(8)-N(9)	107.8(5)
C(8)-N(9)-C(1)	114.0(5)	N(23)-N(9)-C(1)	119.0(5)
N(23)-N(9)-C(8)	116.6(5)	O(11)-N(10)-N(2)	115.6(7)
O(12)-N(10)-N(2)	118.8(7)	O(12)-N(10)-O(11)	125.6(6)
O(14)-N(13)-N(5)	116.5(6)	O(15)-N(13)-N(5)	116.4(7)
O(15)-N(13)-O(14)	126.9(6)	O(17)-N(16)-N(7)	116.6(5)
O(18)-N(16)-N(7)	117.5(6)	O(18)-N(16)-O(17)	125.9(5)
C(20)-O(19)-C(8)	116.7(6)	O(21)-C(20)-O(19)	123.3(7)
C(22)-C(20)-O(19)	107.5(8)	C(22)-C(20)-O(21)	129.2(7)
O(24)-N(23)-N(9)	114.7(5)	O(25)-N(23)-N(9)	116.5(5)
O(25)-N(23)-O(24)	128.6(5)	C(6')-C(1')-N(2')	112.7(5)
N(9')-C(1')-N(2')	111.2(5)	N(9')-C(1')-C(6')	102.0(5)
C(3')-N(2')-C(1')	114.6(5)	N(10')-N(2')-C(1')	118.5(6)
N(10')-N(2')-C(3')	117.2(7)	C(4')-C(3')-N(2')	111.9(7)
N(5')-C(4')-C(3')	109.5(7)	C(6')-N(5')-C(4')	124.3(7)
N(13')-N(5')-C(4')	118.6(7)	N(13')-N(5')-C(6')	117.1(6)
N(5')-C(6')-C(1')	109.8(5)	N(7')-C(6')-C(1')	102.7(5)
N(7')-C(6')-N(5')	114.2(5)	C(8')-N(7')-C(6')	116.3(5)
N(16')-N(7')-C(6')	120.1(7)	N(16')-N(7')-C(8')	116.1(7)
N(9')-C(8')-N(7')	99.3(5)	O(19')-C(8')-N(7')	108.7(5)
O(19')-C(8')-N(9')	110.9(5)	C(8')-N(9')-C(1')	117.1(5)
N(23')-N(9')-C(1')	121.5(5)	N(23')-N(9')-C(8')	121.3(6)
O(11')-N(10')-N(2')	117.0(7)	O(12')-N(10')-N(2')	115.3(8)
O(12')-N(10')-O(11')	127.7(8)	O(14')-N(13')-N(5')	116.2(9)
O(15')-N(13')-N(5')	120.5(7)	O(15')-N(13')-O(14')	123.2(10)
O(17')-N(16')-N(7')	116.4(8)	O(18')-N(16')-N(7')	114.9(8)
O(18')-N(16')-O(17')	128.5(7)	C(20')-O(19')-C(8')	119.7(5)
O(21')-C(20')-O(19')	121.6(7)	C(22')-C(20')-O(19')	111.4(6)
C(22')-C(20')-O(21')	127.0(8)	O(24')-N(23')-N(9')	116.9(5)
O(25')-N(23')-N(9')	118.0(6)	O(25')-N(23')-O(24')	125.2(6)

Abstract

1,4,6,9-Tetranitro-1,4,6,9-tetraaza-5,10-dioxaperhydroanthracene, $C_8H_{12}N_8O_{10}$, M_r = 360.23, triclinic, $P\bar{1}$, a = 7.154(1), b = 10.011(2), c = 11.081(2) Å, α = 66.43(1) , β = 79.06(1), γ = 79.93(1), V = 709.8(2) $Å^3$, Z = 2, D_x = 1.779 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 13.92 cm^{-1}, F(000) = 392, T = 295 K, Final R = 0.033, wR = 0.045, for 2161 independent reflections. There are two molecules in the asymmeteric unit.

Experimental

A clear colorless 0.22 x 0.08 x 0.10 mm data crystal was provided by M. Chaykovsky of NSWC White Oak. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within $50 \leq 2\theta \leq 70°$ used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.59 $Å^{-1}$, range of hkl : $-8 \leq h \leq 8$, $-11 \leq k \leq 10$, $-12 \leq l \leq 0$. Standards 200, 030, 004, monitored every 60 reflections with random variation of 2.1 % over data collection, $\theta/2\theta$ mode, scan width $(2.0 + \Delta_{\alpha 1 \alpha 2})°$, scan rate a function of count rate (4 °/min. minimum, 30 °/min. maximum), 2491 reflections measured, 2358 unique, R_{int} = 0.010, 2161 observed with $Fo > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorbtion effects . Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. 283 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, isotropic thermal parameters for all H atoms. (Δ/σ) max = 0.18, R = 0.033, wR = 0.045, S = 1.997. Final difference Fourier excursions 0.18 and -0.21 $eÅ^{-3}$. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1h).



TETRA-NITRO PERHYDROANTHRACENE

TABLE 1h. Atom coordinates ($x10^4$) and temperature factors ($A^2 x10^3$)

atom	x/a	y/b	z/c	U_{eq}
C(1)	8294(2)	943(2)	5006(2)	28(1)*
C(2)	9976(2)	1401(2)	3903(2)	27(1)*
N(3)	11097(2)	2304(1)	4178(1)	30(1)*
C(4)	10491(2)	2694(2)	5343(2)	32(1)*
C(5)	8372(2)	3244(2)	5422(2)	35(1)*
N(6)	7341(2)	2192(2)	5279(2)	34(1)*
O(1)	8918(1)	-130(1)	6222(1)	29(1)*
N(7)	12226(2)	3195(1)	3121(1)	33(1)*
O(7a)	13080(2)	4036(1)	3310(1)	46(1)*
O(7b)	12344(2)	3052(1)	2058(1)	46(1)*
N(8)	5395(2)	2273(1)	5640(1)	32(1)*
O(8a)	4666(2)	3290(1)	5983(1)	43(1)*
O(8b)	4553(2)	1353(1)	5578(1)	44(1)*
C(1')	3355(2)	1005(2)	9754(2)	29(1)*
C(2')	4501(2)	823(2)	10848(2)	28(1)*
N(3')	5836(2)	1926(1)	10353(1)	33(1)*
C(4')	5708(3)	3134(2)	9066(2)	39(1)*
C(5')	3657(3)	3716(2)	8858(2)	42(1)*
N(6')	2558(2)	2496(1)	9152(1)	36(1)*
O(1')	4565(2)	625(1)	8718(1)	30(1)*
N(7')	6662(2)	2111(2)	11286(2)	43(1)*
O(7'a)	7707(2)	3091(2)	10902(2)	66(1)*
O(7'b)	6349(2)	1245(2)	12432(1)	60(1)*
N(8')	1012(2)	2741(2)	8495(2)	44(1)*
O(8'a)	504(2)	4021(2)	7833(2)	63(1)*
O(8'b)	239(2)	1681(2)	8628(2)	59(1)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

TABLE 2h. Bond lengths (A)

C(1)-C(2)	1.529(2)	C(1)-N(6)	1.430(2)
C(1)-O(1)	1.439(2)	C(2)-N(3)	1.456(3)
C(2)-O(1a)	1.418(2)	N(3)-C(4)	1.462(3)
N(3)-N(7)	1.371(2)	C(4)-C(5)	1.518(2)
C(5)-N(6)	1.459(3)	N(6)-N(8)	1.371(2)
O(1)-C(2a)	1.418(2)	N(7)-O(7a)	1.222(2)
N(7)-O(7b)	1.228(2)	N(8)-O(8a)	1.225(2)
N(8)-O(8b)	1.215(2)	C(1')-C(2')	1.525(3)
C(1')-N(6')	1.431(2)	C(1')-O(1')	1.438(2)
C(2')-N(3')	1.454(2)	C(2')-O(1'a)	1.416(2)
N(3')-C(4')	1.461(2)	N(3')-N(7')	1.370(3)
C(4')-C(5')	1.506(3)	C(5')-N(6')	1.461(3)
N(6')-N(8')	1.371(2)	O(1')-C(2'a)	1.416(2)
N(7')-O(7'a)	1.226(2)	N(7')-O(7'b)	1.222(2)
N(8')-O(8'a)	1.224(2)	N(8')-O(8'b)	1.226(3)

TABLE 3h. Bond angles (deg.)

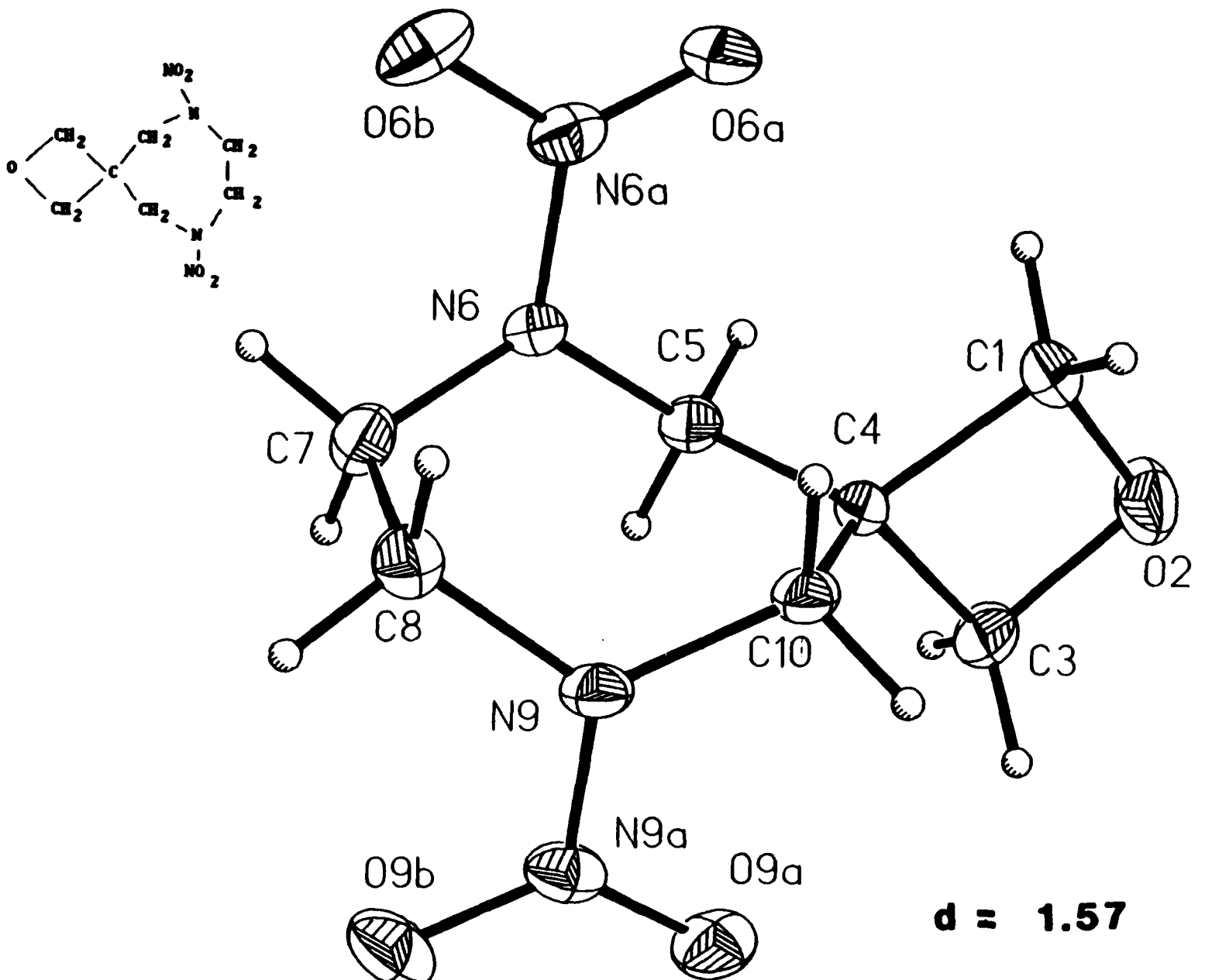
C(2)-C(1)-N(6)	109.6(1)	C(2)-C(1)-O(1)	111.8(1)
N(6)-C(1)-O(1)	107.5(1)	C(1)-C(2)-N(3)	109.9(1)
C(1)-C(2)-O(1a)	109.2(1)	N(3)-C(2)-O(1a)	112.5(1)
C(2)-N(3)-C(4)	120.2(1)	C(2)-N(3)-N(7)	116.9(1)
C(4)-N(3)-N(7)	118.5(1)	N(3)-C(4)-C(5)	110.0(2)
C(4)-C(5)-N(6)	108.3(1)	C(1)-N(6)-C(5)	122.7(1)
C(1)-N(6)-N(8)	118.5(1)	C(5)-N(6)-N(8)	117.9(1)
C(1)-O(1)-C(2a)	114.6(1)	N(3)-N(7)-O(7a)	118.1(2)
N(3)-N(7)-O(7b)	116.9(2)	O(7a)-N(7)-O(7b)	125.0(1)
N(6)-N(8)-O(8a)	116.2(2)	N(6)-N(8)-O(8b)	117.7(1)
O(8a)-N(8)-O(8b)	126.1(1)	C(2')-C(1')-N(6')	110.9(2)
C(2')-C(1')-O(1')	110.7(1)	N(6')-C(1')-O(1')	107.0(1)
C(1')-C(2')-N(3')	109.8(1)	C(1')-C(2')-O(1'a)	107.7(1)
N(3')-C(2')-O(1'a)	112.6(1)	C(2')-N(3')-C(4')	120.3(1)
C(2')-N(3')-N(7')	116.4(1)	C(4')-N(3')-N(7')	118.8(1)
N(3')-C(4')-C(5')	111.4(1)	C(4')-C(5')-N(6')	109.4(1)
C(1')-N(6')-C(5')	121.4(1)	C(1')-N(6')-N(8')	116.9(2)
C(5')-N(6')-N(8')	118.7(1)	C(1')-O(1')-C(2'a)	114.4(1)
N(3')-N(7')-O(7'a)	117.8(1)	N(3')-N(7')-O(7'b)	116.9(2)
O(7'a)-N(7')-O(7'b)	125.3(2)	N(6')-N(8')-O(8'a)	116.4(2)
N(6')-N(8')-O(8'b)	118.2(1)	O(8'a)-N(8')-O(8'b)	125.4(2)

Abstract

2-Oxa-6,9-diaza-6,9-dinitrospiro[3.6]decane, $C_7H_{12}N_4O_5$, M_r = 232.20, triclinic, $P\bar{1}$, a = 6.028(1), b = 6.420(2), c = 13.674(40 Å, α = 91.04(20, β = 100.22(2) γ = 108.57(2)°, V = 492.0(20 Å^3, Z = 2, D_x = 1.567 Mg m^{-3}, λ(Cu Kα) = 1.54178Å, μ = 11.08 cm^{-1}, F(000) = 244, T = 295 K, Final R = 0.075, wR = 0.075, for 877 independent reflections.

Experimental

A clear colorless 0.02 x 0.08 x 0.15 mm data crystal recrystallized from ethylacetate was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet R3m diffractometer with incident beam monochromator. 1864 centered reflections within $32 \leq 2\theta \leq 75°$ used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.55 Å^{-1}, range of hkl: $0 \leq h \leq 6$, $-7 \leq k \leq 7$, $-15 \leq l \leq 15$. Standards 021, 200, 003, monitored every 60 reflections with linear variation of 4.8% over data collection, $\theta/2\theta$ mode, scan width $(2.0 + \Delta_{\alpha 1 \alpha 2})°$, scan rate a function of count rate (3.0°/min. minimum, 30.0°/min. maximum, 1864 reflections measured, 1253 unique, R_{int} = 0.045, 877 observed with $Fo > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorbtion effects. Secondary extinction parameter p = 0.022(5) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin 2\theta]^{0.25}$. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.0006. 146 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model , C-H = 0.96 Å, C-C-H = 109.5°, $U(H) = 1.2\ U_{eq}(C)$. (Δ/σ) max = 0.006, R = 0.075, wR = 0.075, S = 1.644. Final difference Fourier excursions 0.28 and -0.29 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1i).



2-Oxa-6,9-diaza-6,9-dinitrospiro(3.6)decane

TABLE **1i.** Atom coordinates ($x10^4$) and thermal parameters ($Å^2 \times 10^3$)

atom	x	y	z	U
C(1)	164(10)	2834(11)	851(4)	60(3)*
O(2)	1709(8)	3174(8)	142(3)	77(2)*
C(3)	3659(11)	2927(11)	878(4)	61(3)*
C(4)	2085(9)	2447(9)	1688(4)	44(2)*
C(5)	1568(9)	49(9)	1973(4)	46(2)*
N(6)	510(7)	-422(7)	2862(3)	45(2)*
C(7)	1964(11)	-64(9)	3850(4)	57(3)*
C(8)	2906(11)	2350(10)	4210(4)	58(3)*
N(9)	4132(7)	3663(7)	3484(3)	46(2)*
C(10)	2788(9)	4127(8)	2570(4)	46(2)*
N(6a)	-1878(8)	-740(7)	2763(3)	53(2)*
O(6a)	-3023(7)	-790(7)	1931(3)	66(2)*
O(6b)	-2689(8)	-1014(7)	3542(3)	73(2)*
N(9a)	6474(8)	3855(7)	3526(4)	52(2)*
O(9a)	7321(7)	4492(7)	2790(3)	64(2)*
O(9b)	7564(8)	3444(7)	4303(3)	74(2)*

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

TABLE **2i.** Bond lengths (A)

C(1)-O(2)	1.433(8)	C(1)-C(4)	1.561(8)
O(2)-C(3)	1.457(8)	C(3)-C(4)	1.556(8)
C(4)-C(5)	1.542(8)	C(4)-C(10)	1.509(7)
C(5)-N(6)	1.463(7)	N(6)-C(7)	1.445(6)
N(6)-N(6a)	1.369(7)	C(7)-C(8)	1.511(8)
C(8)-N(9)	1.461(7)	N(9)-C(10)	1.454(7)
N(9)-N(9a)	1.367(7)	N(6a)-O(6a)	1.215(6)
N(6a)-O(6b)	1.242(7)	N(9a)-O(9a)	1.227(7)
N(9a)-O(9b)	1.226(7)		

TABLE **3i.** Bond angles (deg.)

O(2)-C(1)-C(4)	92.3(5)	C(1)-O(2)-C(3)	92.2(4)
O(2)-C(3)-C(4)	91.6(5)	C(1)-C(4)-C(3)	83.8(4)
C(1)-C(4)-C(5)	115.2(4)	C(3)-C(4)-C(5)	111.1(5)
C(1)-C(4)-C(10)	112.2(5)	C(3)-C(4)-C(10)	117.2(4)
C(5)-C(4)-C(10)	114.0(4)	C(4)-C(5)-N(6)	114.7(5)
C(5)-N(6)-C(7)	121.7(4)	C(5)-N(6)-N(6a)	117.8(4)
C(7)-N(6)-N(6a)	119.0(5)	N(6)-C(7)-C(8)	111.3(5)
C(7)-C(8)-N(9)	110.6(5)	C(8)-N(9)-C(10)	120.7(4)
C(8)-N(9)-N(9a)	117.5(5)	C(10)-N(9)-N(9a)	118.8(4)
C(4)-C(10)-N(9)	117.3(5)	N(6)-N(6a)-O(6a)	118.1(5)
N(6)-N(6a)-O(6b)	116.0(4)	O(6a)-N(6a)-O(6b)	125.8(5)
N(9)-N(9a)-O(9a)	116.8(5)	N(9)-N(9a)-O(9b)	117.5(5)
O(9a)-N(9a)-O(9b)	125.6(5)		

Abstract

8,11-Dibromo-8,11-dinitro-pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane, $C_{11}H_{10}N_2O_4Br_2$, M_r = 394.03, monoclinic, $P2_1$, a = 6.788(2), b = 13.696(4), c = 6.931(2) Å, β =107.19(2)°, V = 615.5(3) Å^3, Z = 2, D_x = 2.126 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 85.9 cm^{-1}, F(000) = 384, T = 295 K, Final R = 0.041, wR = 0.050, for 1003 independent reflections.

Experimental

A clear colorless 0.10 x 0.12 x 0.25 mm data crystal was provided by W. W. Zajac of Villanova University. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within 33≤ 2θ ≤78° used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.58 Å^{-1}, range of hkl : -7 ≤ h ≤ 0, 0 ≤ k ≤ 15, -7 ≤ ≤ 7. Standards 200, 060, 003, monitored every 60 reflections with random variation of 2.2 % over data collection, θ/2θ mode, scan width [2θ(Kα$_1$) - 1.0] to [2θ(Kα$_2$) + 1.0]°, scan rate a function of count rate (10°/min. minimum, 30°/min. maximum, 1188 reflections measured, 1030 unique, R_{int} = 0.029, 1003 observed with Fo>3σ(F_o). Data corrected for Lorentz , polarization, and absorbtion effects , max and min trans 0.86 and 0.46. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. 171 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model , C-H = 0.96 Å, C-C-H = 109.5° , U(H) = 1.1· U_{eq}(C) . (Δ/σ) max = 0.005, R = 0.041, wR = 0.050, S = 2.133. Final difference Fourier excursions 0.84 and -0.44 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1j).

8,11-Dibromo-8,11-dinitro-pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane

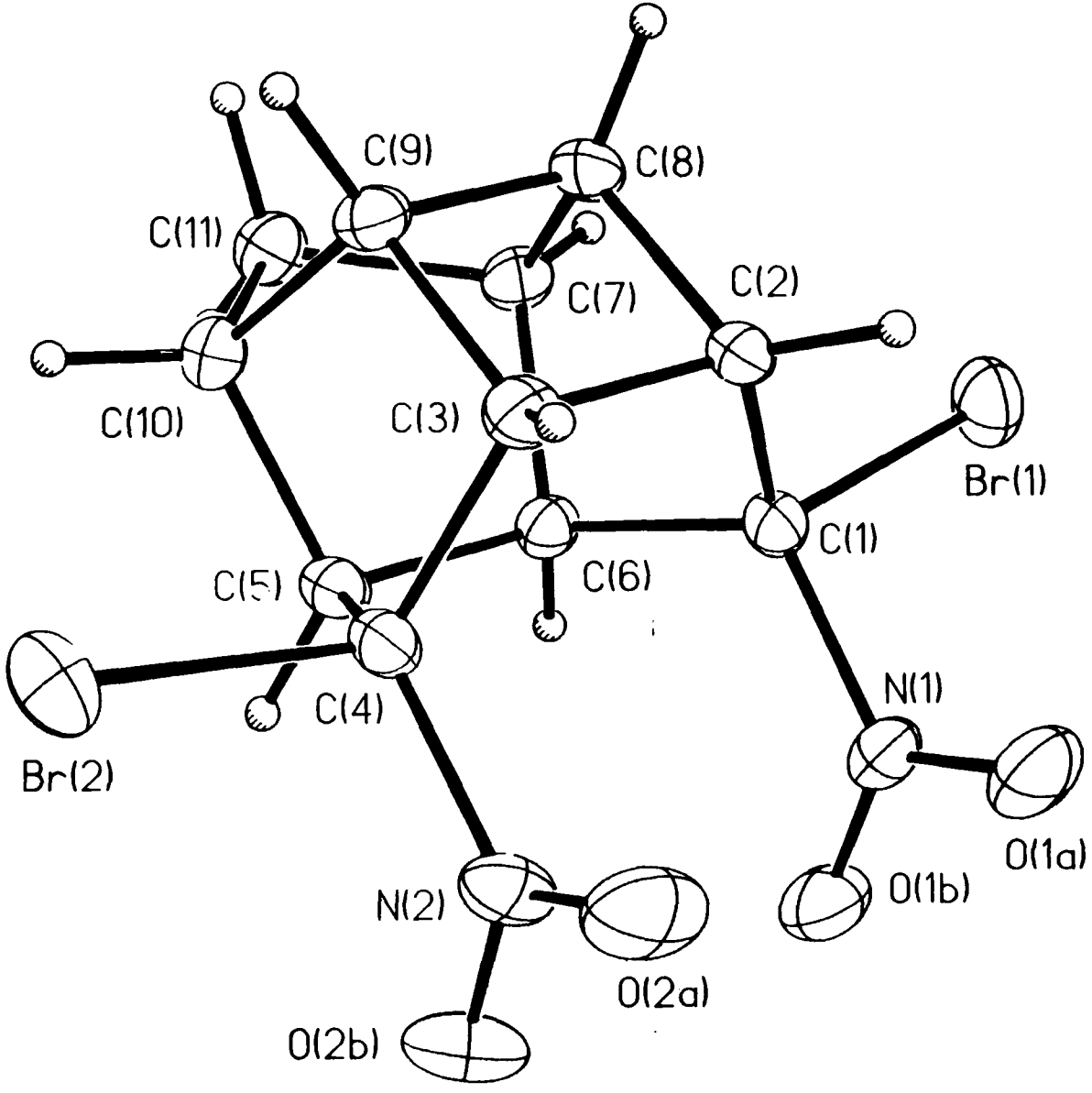


TABLE **1j.** Atom coordinates ($x10^4$) and thermal parameters ($Å^2 \times 10^3$)

atom	x	y	z	U
C(1)	2097(12)	10628(6)	-959(12)	41(3)*
C(2)	683(12)	10388(6)	335(13)	43(3)*
C(3)	1593(12)	9667(6)	2100(13)	44(3)*
C(4)	3758(12)	9308(6)	2310(12)	41(3)*
C(5)	3527(12)	8882(6)	223(12)	41(3)*
C(6)	2615(12)	9615(6)	-1590(12)	39(3)*
C(7)	387(13)	9212(6)	-2528(13)	45(3)*
C(8)	-643(12)	9541(7)	-930(13)	45(3)*
C(9)	241(12)	8827(7)	807(12)	43(3)*
C(10)	1691(12)	8174(7)	31(13)	46(3)*
C(11)	614(15)	8108(7)	-2189(14)	54(3)*
Br(1)	555(2)	11370	-3331(2)	66(1)*
N(1)	3880(12)	11307(7)	-90(12)	51(3)*
O(1a)	3647(12)	11934(6)	1047(15)	83(4)*
O(1b)	5415(12)	11187(6)	-569(13)	71(3)*
Br(2)	4534(2)	8246(1)	4374(2)	67(1)*
N(2)	5488(11)	10026(7)	3150(10)	52(3)*
O(2a)	5242(13)	10621(7)	4327(14)	88(4)*
O(2b)	7032(10)	9944(7)	2617(11)	75(3)*

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

TABLE **2j.** Bond lengtns (A)

C(1)-C(2)	1.531(14)	C(1)-C(6)	1.527(12)
C(1)-Br(1)	1.953(8)	C(1)-N(1)	1.503(11)
C(2)-C(3)	1.551(12)	C(2)-C(8)	1.568(12)
C(3)-C(4)	1.516(12)	C(3)-C(9)	1.576(11)
C(4)-C(5)	1.524(12)	C(4)-Br(2)	1.998(8)
C(4)-N(2)	1.510(11)	C(5)-C(6)	1.584(11)
C(5)-C(10)	1.553(12)	C(6)-C(7)	1.560(11)
C(7)-C(8)	1.542(14)	C(7)-C(11)	1.531(12)
C(8)-C(9)	1.529(12)	C(9)-C(10)	1.539(13)
C(10)-C(11)	1.498(12)	N(1)-O(1a)	1.207(13)
N(1)-O(1b)	1.195(13)	N(2)-O(2a)	1.200(13)
N(2)-O(2b)	1.215(11)		

TABLE **3j.** Bond angles (deg.)

C(2)-C(1)-C(6)	102.2(7)	C(2)-C(1)-Br(1)	109.4(5)
C(6)-C(1)-Br(1)	110.1(5)	C(2)-C(1)-N(1)	117.9(7)
C(6)-C(1)-N(1)	116.8(7)	Br(1)-C(1)-N(1)	100.6(5)
C(1)-C(2)-C(3)	115.2(7)	C(1)-C(2)-C(8)	100.8(7)
C(3)-C(2)-C(8)	89.9(6)	C(2)-C(3)-C(4)	115.5(8)
C(2)-C(3)-C(9)	89.3(6)	C(4)-C(3)-C(9)	101.7(7)
C(3)-C(4)-C(5)	102.0(6)	C(3)-C(4)-Br(2)	110.4(6)
C(5)-C(4)-Br(2)	109.4(6)	C(3)-C(4)-N(2)	116.5(7)
C(5)-C(4)-N(2)	117.7(8)	Br(2)-C(4)-N(2)	100.8(5)
C(4)-C(5)-C(6)	114.6(7)	C(4)-C(5)-C(10)	100.1(7)
C(6)-C(5)-C(10)	102.2(6)	C(1)-C(6)-C(5)	114.7(7)
C(1)-C(6)-C(7)	99.4(6)	C(5)-C(6)-C(7)	102.1(7)
C(6)-C(7)-C(8)	100.8(6)	C(6)-C(7)-C(11)	104.1(6)
C(8)-C(7)-C(11)	103.0(8)	C(2)-C(8)-C(7)	108.1(7)
C(2)-C(8)-C(9)	90.4(6)	C(7)-C(8)-C(9)	102.9(7)
C(3)-C(9)-C(8)	90.4(6)	C(3)-C(9)-C(10)	107.3(7)
C(8)-C(9)-C(10)	103.8(7)	C(5)-C(10)-C(9)	100.8(7)
C(5)-C(10)-C(11)	105.1(8)	C(9)-C(10)-C(11)	103.1(7)
C(7)-C(11)-C(10)	95.4(7)	C(1)-N(1)-O(1a)	117.2(8)
C(1)-N(1)-O(1b)	118.0(8)	O(1a)-N(1)-O(1b)	124.9(9)
C(4)-N(2)-O(2a)	117.1(8)	C(4)-N(2)-O(2b)	117.7(8)
O(2a)-N(2)-O(2b)	125.1(9)		

Abstract

8-Bromo-8-nitro-pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-11-one, $C_{11}H_{10}N_1O_3Br$, M_r = 284.11, monoclinic, $P2_1/n$, a = 11.155(3), b = 8.180(2), c = 12.387 Å, β = 113.55(2)°, V =1036.1(9) Å^3, Z = 4, D_x = 1.821 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 53.9 cm^{-1}, F(000) = 568, T = 295 K, Final R = 0.038, wR = 0.050 for 1384 independent reflections.

Experimental

A clear colorless 0.07 x 0.30 x 0.35 mm data crystal was provided by W. W. Zajac of Villanova University. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within 34≤ 2θ ≤60° used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.58 Å^{-1}, range of hkl : 0 ≤ h ≤ 12, 0 ≤ k ≤ 8, -12 ≤ ≤ 12. Standards 400, 040, 006, monitored every 60 reflections with linear variation of 2.7 % over data collection, θ/2θ mode, scan width [2θ(Kα$_1$) - 1.0] to [2θ(Kα$_2$) + 1.0]°, scan rate a function of count rate (5.0°/min. minimum, 30°/min. maximum, 1759 reflections measured, 1461 unique, R_{int} = 0.01, 1384 observed with Fo>3σ(F_o). Data corrected for Lorentz , polarization, and absorbtion effects , max and min trans 0.81 and 0.46. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. 145 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model , C-H = 0.96 Å, C-C-H = 109.5°, U(H) = 1.1·U_{eq}(C) . (Δ/σ) max = 0.003, R = 0.038, wR = 0.050, S = 2.291. Final difference Fourier excursions 0.33 and -0.38 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1k).

8-Bromo-8-nitro-pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-11-one

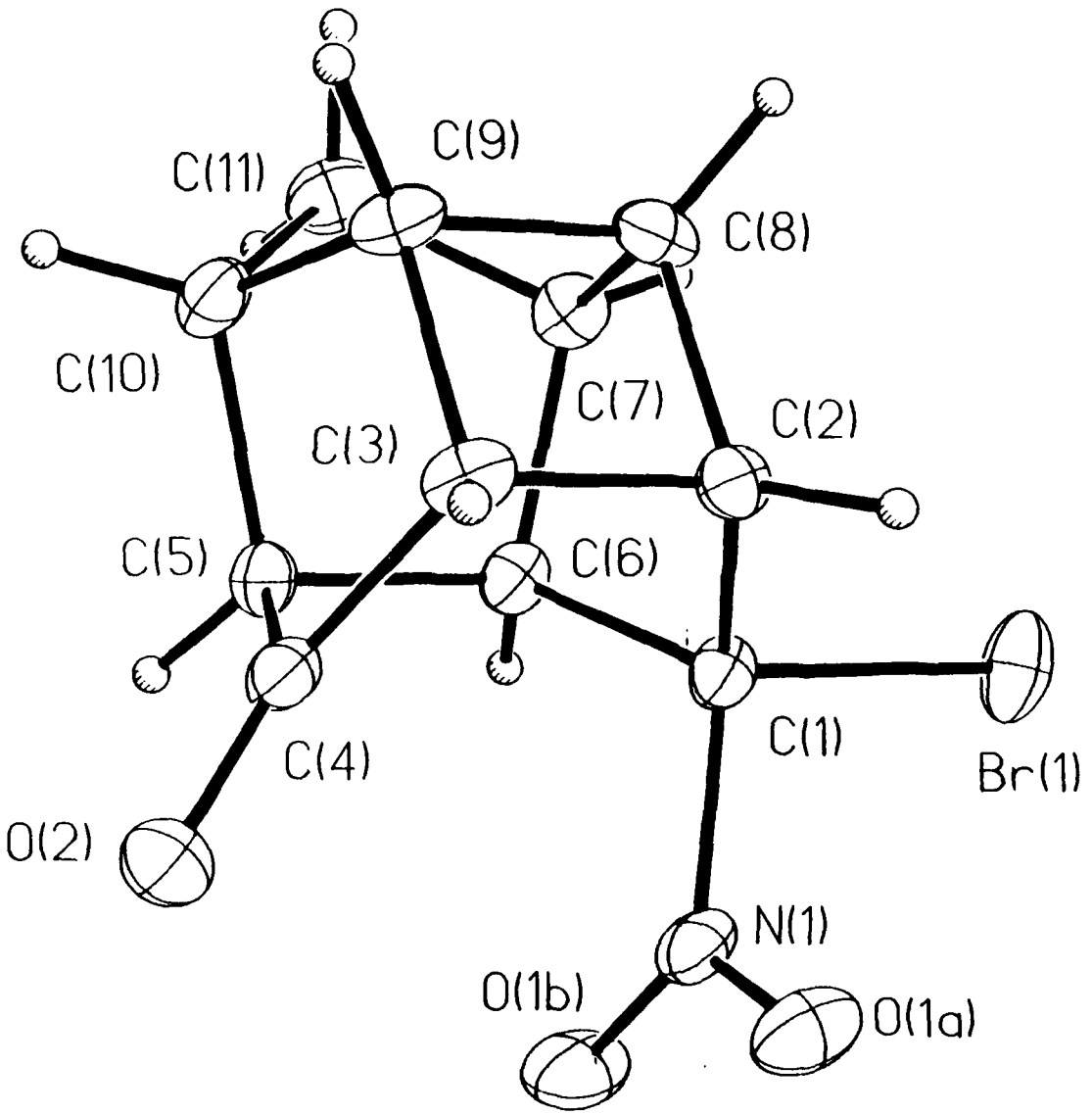


Table 1k. Atom coordinates ($x10^4$) and thermal parameters ($Å^2 \times 10^3$)

atom	x	y	z	U
C(1)	666(3)	5373(4)	3015(3)	37(1)*
C(2)	1194(3)	4171(4)	2384(3)	37(1)*
C(3)	88(4)	3448(4)	1259(3)	42(1)*
C(4)	-1216(3)	4130(4)	1107(3)	41(1)*
C(5)	-1388(3)	3610(4)	2210(3)	40(1)*
C(6)	-266(3)	4327(4)	3346(3)	37(1)*
C(7)	568(3)	2803(4)	3896(3)	43(1)*
C(8)	1268(3)	2551(4)	3057(3)	43(1)*
C(9)	165(4)	1823(4)	1937(3)	45(1)*
C(10)	-1019(4)	1772(4)	2284(3)	46(1)*
C(11)	-413(4)	1405(4)	3592(4)	54(2)*
Br(1)	2072(1)	6269(1)	4401(1)	66(1)*
N(1)	34(3)	6885(3)	2297(3)	45(1)*
O(1a)	501(3)	7431(3)	1641(3)	63(1)*
O(1b)	-902(3)	7457(3)	2432(3)	73(1)*
O(2)	-1983(3)	4858(3)	274(2)	59(1)*

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

TABLE **2k.** Bond lengths (A)

C(1)-C(2)	1.514(5)	C(1)-C(6)	1.523(5)
C(1)-Br(1)	1.948(3)	C(1)-N(1)	1.523(4)
C(2)-C(3)	1.562(4)	C(2)-C(8)	1.550(5)
C(3)-C(4)	1.498(5)	C(3)-C(9)	1.558(5)
C(4)-C(5)	1.515(6)	C(4)-O(2)	1.203(4)
C(5)-C(6)	1.576(4)	C(5)-C(10)	1.551(4)
C(6)-C(7)	1.543(4)	C(7)-C(8)	1.543(6)
C(7)-C(11)	1.522(5)	C(8)-C(9)	1.559(4)
C(9)-C(10)	1.543(6)	C(10)-C(11)	1.516(5)
N(1)-O(1a)	1.212(5)	N(1)-O(1b)	1.215(5)

TABLE **3k.** Bond angles (deg.)

C(2)-C(1)-C(6)	102.9(3)	C(2)-C(1)-Br(1)	110.9(2)
C(6)-C(1)-Br(1)	111.7(2)	C(2)-C(1)-N(1)	114.3(3)
C(6)-C(1)-N(1)	114.0(3)	Br(1)-C(1)-N(1)	103.4(2)
C(1)-C(2)-C(3)	112.0(3)	C(1)-C(2)-C(8)	103.1(3)
C(3)-C(2)-C(8)	90.[illegible]2)	C(2)-C(3)-C(4)	110.6(3)
C(2)-C(3)-C(9)	89.3(2)	C(4)-C(3)-C(9)	103.1(3)
C(3)-C(4)-C(5)	104.4(3)	C(3)-C(4)-O(2)	127.9(4)
C(5)-C(4)-O(2)	127.5(4)	C(4)-C(5)-C(6)	111.0(3)
C(4)-C(5)-C(10)	101.5(3)	C(6)-C(5)-C(10)	102.2(2)
C(1)-C(6)-C(5)	110.7(3)	C(1)-C(6)-C(7)	102.5(3)
C(5)-C(6)-C(7)	102.5(2)	C(6)-C(7)-C(8)	100.9(3)
C(6)-C(7)-C(11)	104.5(3)	C(8)-C(7)-C(11)	103.9(3)
C(2)-C(8)-C(7)	108.4(3)	C(2)-C(8)-C(9)	89.7(2)
C(7)-C(8)-C(9)	102.6(3)	C(3)-C(9)-C(8)	90.4(2)
C(3)-C(9)-C(10)	107.7(3)	C(8)-C(9)-C(10)	102.8(3)
C(5)-C(10)-C(9)	101.2(3)	C(5)-C(10)-C(11)	104.4(3)
C(9)-C(10)-C(11)	103.7(3)	C(7)-C(11)-C(10)	94.9(3)
C(1)-N(1)-O(1a)	117.9(3)	C(1)-N(1)-O(1b)	117.1(3)
O(1a)-N(1)-O(1b)	125.0(3)		

Abstract

3,6-Dihydroxy-2,7-diethoxycarbonyl pentacyclo[6.5.0.0^{1,8}.0^{4,5}.0^{9,13}]trideca-2,6-diene, $C_{19}H_{22}O_6$, M_r = 346.38, monoclinic, $P2_1/c$, a = 11.028(4), b = 20.429(6), c = 16.015(3) Å, β = 105.28(3)°, V = 3480.5(2) Å^3, Z = 8 (two molecules per asymmetric unit), D_x = 1.32 Mg m^{-3}, λ(Mo Kα) = 0.71069 Å, μ = 5.91 cm^{-1}, F(000) = 1472, T = 295 K, Final R = 0.065, wR = 0.056 for 3172 independent reflections. Each of the two molecules in the asymmetric unit is similarly configured. Bond lengths C(1)-C(8), 1.575(5) and1.572(5), and C(4)-C(5), 1.582(5) and 1.586(5) Å are longer than normal and the C(10)-C(11)-C(12) angle is 94.7(3) and 94.8(3)°, which is much less than the normal tetrahedral angle. These deviations are due to internal cage strains and repulsive forces between ethoxycarbonyl and hydroxyl groups. There are two intramolecular hydrogen bonds per molecule.

Experimental

A clear colorless data crystal was provided by A. Marchand of North Texas State University. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within 18 ≤ 2θ ≤ 31° used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.65 Å^{-1}, range of hkl : 0 ≤ h ≤ 11, 0 ≤ k ≤ 26, -20 ≤ ≤ 20. Standards 400, 080, 004, monitored every 60 reflections with random variation of 1.9 % over data collection, θ/2θ mode, scan width [2θ(Kα$_1$) - 0.7] to [2θ(Kα$_2$) + 0.7]°, scan rate a function of count rate (8.0°/min. minimum, 30.0°/min. maximum, 5668 reflections measured, 4796 unique, R_{int} = 0.008, 3172 observed with Fo>3σ(F_o). Data corrected for Lorentz and polarization, but not absorbtion effects . Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. 476 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model (coordinate shifts of C applied to attached H atoms), C-H = 0.96 Å, U(H) = 1.2 U_{eq}(C) . (Δ/σ) max = 0.31, R = 0.065, wR = 0.056. Final difference Fourier excursions 0.26 and -

0.23 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(11).

6-Dihydroxy-2,7-diethoxycarbonyl pentacyclo[6.5.0.0^{1,8}.0^{4,5}.0^{9,13}]tri-deca-2,6-diene

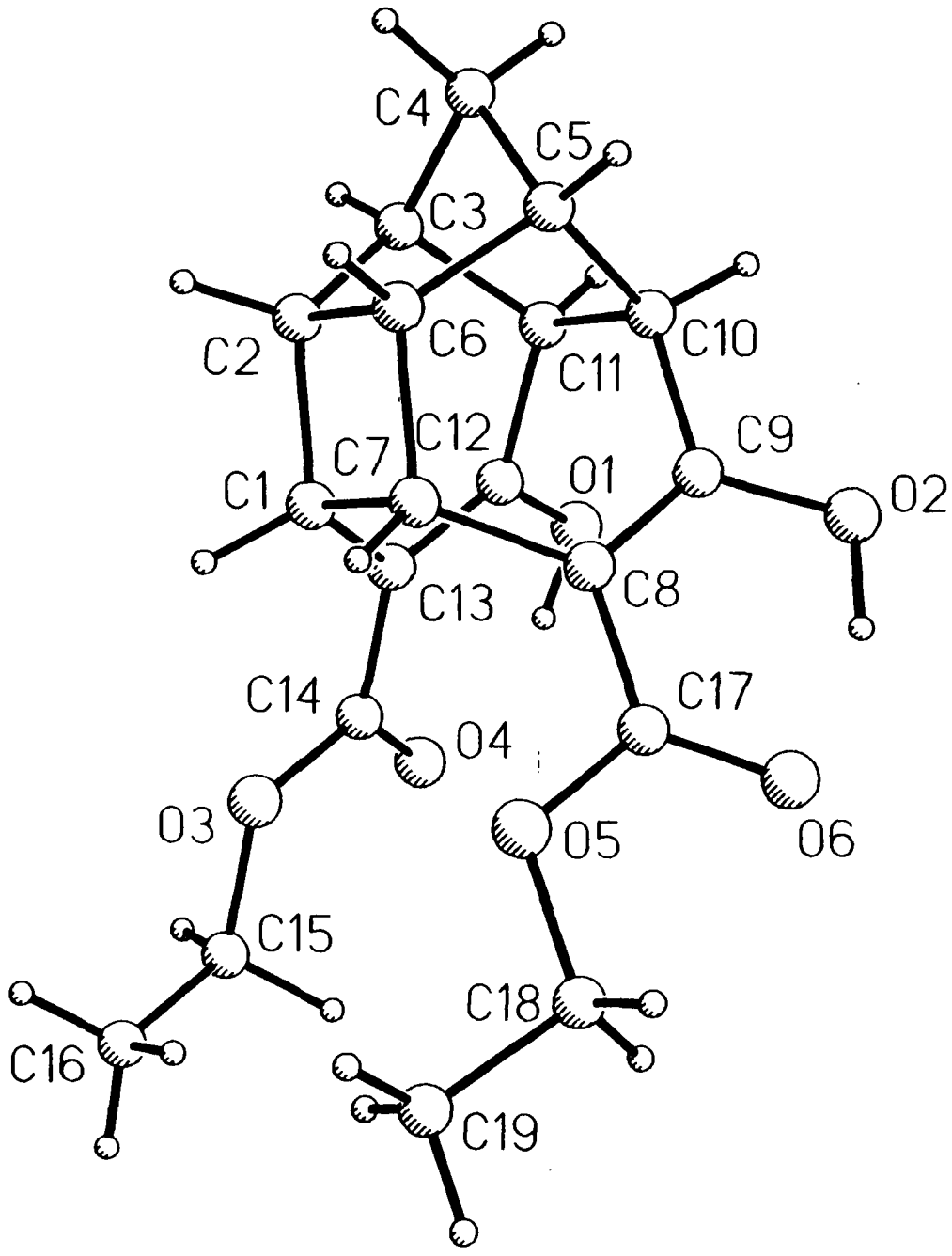


Table 11. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2 x 10^3$)

	x	y	z	U(eq)
O(1)	-.5040(3)	0.4047(2)	0.0711(2)	75(2)
O(2)	-.6552(3)	0.4077(2)	-.1819(2)	80(2)
O(3)	-.4191(3)	0.2083(2)	0.0594(2)	62(1)
O(4)	-.5161(3)	0.2851(2)	0.1189(2)	75(1)
O(5)	-.5786(3)	0.2108(2)	-.1944(2)	58(1)
O(6)	-.7225(3)	0.2902(2)	-.2256(2)	82(2)
C(1)	-.3124(3)	0.2984(2)	-.0340(2)	44(2)
C(2)	-.3948(4)	0.3177(2)	0.0233(2)	46(2)
C(3)	-.4265(4)	0.3810(2)	0.0251(3)	55(2)
C(4)	-.3764(4)	0.4316(2)	-.0239(3)	58(2)
C(5)	-.4382(4)	0.4323(2)	-.1250(3)	57(2)
C(6)	-.5388(4)	0.3830(2)	-.1584(3)	54(2)
C(7)	-.5100(4)	0.3192(2)	-.1650(2)	46(2)
C(8)	-.3747(4)	0.2991(2)	-.1345(2)	44(2)
C(9)	-.2781(4)	0.3516(2)	-.1444(2)	49(2)
C(10)	-.3244(4)	0.4224(2)	-.1616(3)	59(2)
C(11)	-.2218(4)	0.4599(2)	-.0969(3)	69(2)
C(12)	-.2362(4)	0.4215(2)	-.0182(3)	58(2)
C(13)	-.2176(4)	0.3507(2)	-.0463(2)	50(2)
C(14)	-.4486(4)	0.2703(2)	0.0715(3)	53(2)
C(15)	-.4739(5)	0.1584(2)	0.1031(3)	84(2)
C(16)	-.4463(7)	0.0945(3)	0.0703(4)	126(4)
C(17)	-.6124(5)	0.2732(2)	-.1976(3)	55(2)
C(18)	-.6804(5)	0.1652(3)	-.2299(3)	84(2)
C(19)	-.6340(5)	0.0987(2)	-.2163(4)	109(3)
O(21)	0.0104(3)	0.4510(2)	0.1901(2)	82(2)
O(22)	0.1507(3)	0.4353(2)	0.4397(2)	86(2)
O(23)	-.0587(3)	0.2520(2)	0.1729(2)	69(1)
O(24)	0.0403(3)	0.3364(2)	0.1301(2)	77(2)
O(25)	0.0881(3)	0.2353(2)	0.4329(2)	63(1)
O(26)	0.2283(3)	0.3167(2)	0.4682(2)	86(2)
C(21)	-.1760(3)	0.3269(2)	0.2727(2)	41(2)
C(22)	-.0923(4)	0.3554(2)	0.2219(2)	47(2)
C(23)	-.0668(4)	0.4193(2)	0.2294(3)	56(2)
C(24)	-.1268(4)	0.4629(2)	0.2822(3)	60(2)
C(25)	-.0696(5)	0.4566(2)	0.3837(3)	64(2)
C(26)	0.0356(4)	0.4081(2)	0.4112(3)	58(2)
C(27)	0.0137(4)	0.3431(2)	0.4094(2)	50(2)
C(28)	-.1198(4)	0.3209(2)	0.3734(2)	45(2)
C(29)	-.2221(4)	0.3679(2)	0.3868(3)	52(2)
C(30)	-.1838(5)	0.4382(2)	0.4151(3)	63(2)
C(31)	-.2880(5)	0.4771(2)	0.3532(3)	79(2)
C(32)	-.2652(4)	0.4472(2)	0.2711(3)	57(2)
C(33)	-.2775(4)	0.3737(2)	0.2889(2)	49(2)
C(34)	-.0318(4)	0.3152(3)	0.1711(3)	59(2)
C(35)	0.0000(5)	0.2080(3)	0.1243(3)	96(3)
C(36)	-.0250(8)	0.1418(3)	0.1471(4)	145(4)
C(37)	0.1192(5)	0.2988(3)	0.4391(3)	57(2)
C(38)	0.1904(4)	0.1892(3)	0.4658(3)	83(2)
C(39)	0.1385(5)	0.1221(3)	0.4448(4)	103(3)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2l. Bond lengths (Å)

O(1)-C(3)	1.355(4)	O(2)-C(6)	1.339(4)
O(3)-C(14)	1.336(4)	O(3)-C(15)	1.455(4)
O(4)-C(14)	1.232(4)	O(5)-C(17)	1.327(4)
O(5)-C(18)	1.454(4)	O(6)-C(17)	1.229(4)
C(1)-C(2)	1.504(5)	C(1)-C(8)	1.575(5)
C(1)-C(13)	1.544(5)	C(2)-C(3)	1.343(5)
C(2)-C(14)	1.458(5)	C(3)-C(4)	1.489(5)
C(4)-C(5)	1.582(5)	C(4)-C(12)	1.538(5)
C(5)-C(6)	1.490(5)	C(5)-C(10)	1.533(5)
C(6)-C(7)	1.352(5)	C(7)-C(8)	1.500(5)
C(7)-C(17)	1.455(5)	C(8)-C(9)	1.547(5)
C(9)-C(10)	1.535(5)	C(9)-C(13)	1.536(5)
C(10)-C(11)	1.525(5)	C(11)-C(12)	1.529(5)
C(12)-C(13)	1.544(5)	C(15)-C(16)	1.470(6)
C(18)-C(19)	1.448(6)	O(21)-C(23)	1.350(4)
O(22)-C(26)	1.350(5)	O(23)-C(34)	1.328(5)
O(23)-C(35)	1.448(5)	O(24)-C(34)	1.235(5)
O(25)-C(37)	1.339(5)	O(25)-C(38)	1.456(4)
O(26)-C(37)	1.226(4)	C(21)-C(22)	1.500(5)
C(21)-C(28)	1.572(5)	C(21)-C(33)	1.544(5)
C(22)-C(23)	1.336(5)	C(22)-C(34)	1.438(5)
C(23)-C(24)	1.496(5)	C(24)-C(25)	1.586(5)
C(24)-C(32)	1.524(5)	C(25)-C(26)	1.500(6)
C(25)-C(30)	1.521(5)	C(26)-C(27)	1.349(5)
C(27)-C(28)	1.503(5)	C(27)-C(37)	1.452(5)
C(28)-C(29)	1.539(5)	C(29)-C(30)	1.532(5)
C(29)-C(33)	1.531(5)	C(30)-C(31)	1.527(5)
C(31)-C(32)	1.529(5)	C(32)-C(33)	1.540(5)
C(35)-C(36)	1.447(6)	C(38)-C(39)	1.489(6)

Table 31. Bond angles (°)

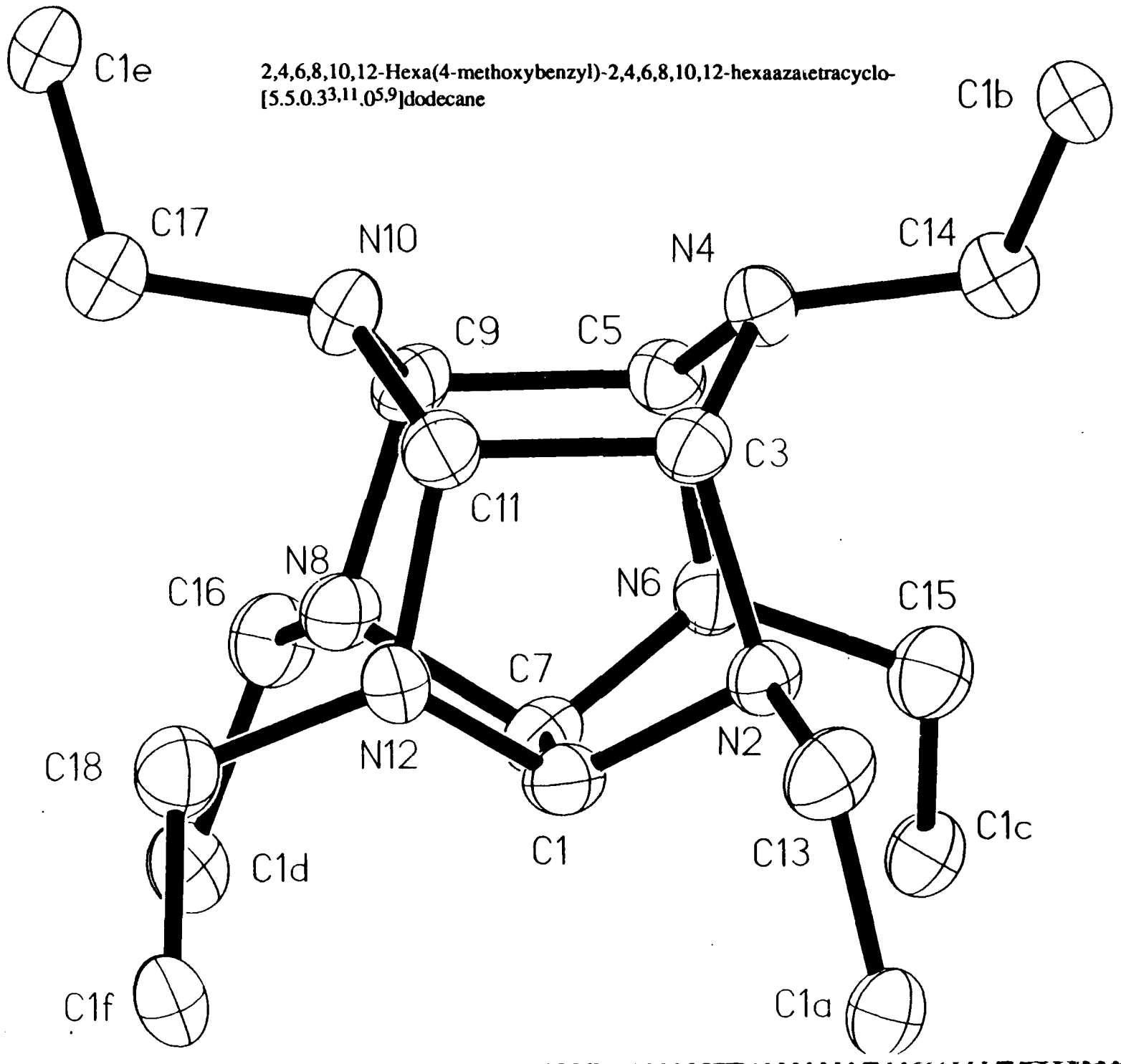
C(15)-O(3)-C(14)	116.5(3)	C(18)-O(5)-C(17)	114.9(3)
C(8)-C(1)-C(2)	116.8(3)	C(13)-C(1)-C(2)	115.7(3)
C(13)-C(1)-C(8)	89.4(3)	C(3)-C(2)-C(1)	118.0(4)
C(14)-C(2)-C(1)	123.0(4)	C(14)-C(2)-C(3)	118.9(4)
C(2)-C(3)-O(1)	124.1(4)	C(4)-C(3)-O(1)	114.4(4)
C(4)-C(3)-C(2)	121.4(4)	C(5)-C(4)-C(3)	115.3(3)
C(12)-C(4)-C(3)	112.5(3)	C(12)-C(4)-C(5)	102.6(3)
C(6)-C(5)-C(4)	116.1(3)	C(10)-C(5)-C(4)	102.4(3)
C(10)-C(5)-C(6)	112.4(3)	C(5)-C(6)-O(2)	114.5(4)
C(7)-C(6)-O(2)	124.7(4)	C(7)-C(6)-C(5)	120.8(4)
C(8)-C(7)-C(6)	118.1(4)	C(17)-C(7)-C(6)	118.3(4)
C(17)-C(7)-C(8)	123.5(4)	C(7)-C(8)-C(1)	117.6(3)
C(9)-C(8)-C(1)	89.1(3)	C(9)-C(8)-C(7)	115.5(3)
C(10)-C(9)-C(8)	117.8(3)	C(13)-C(9)-C(8)	90.7(3)
C(13)-C(9)-C(10)	103.3(3)	C(9)-C(10)-C(5)	108.8(3)
C(11)-C(10)-C(5)	102.1(3)	C(11)-C(10)-C(9)	101.5(3)
C(12)-C(11)-C(10)	94.7(3)	C(11)-C(12)-C(4)	101.8(3)
C(13)-C(12)-C(4)	108.4(3)	C(13)-C(12)-C(11)	100.9(3)
C(9)-C(13)-C(1)	90.7(3)	C(12)-C(13)-C(1)	117.7(3)
C(12)-C(13)-C(9)	103.4(3)	O(4)-C(14)-O(3)	122.1(4)
C(2)-C(14)-O(3)	113.8(4)	C(2)-C(14)-O(4)	124.1(4)
C(16)-C(15)-O(3)	107.4(4)	O(6)-C(17)-O(5)	121.7(4)
C(7)-C(17)-O(5)	115.1(4)	C(7)-C(17)-O(6)	123.2(4)
C(19)-C(18)-O(5)	109.6(4)	C(35)-O(23)-C(34)	117.2(4)
C(38)-O(25)-C(37)	116.1(3)	C(28)-C(21)-C(22)	117.0(3)
C(33)-C(21)-C(22)	115.3(3)	C(33)-C(21)-C(28)	89.1(3)
C(23)-C(22)-C(21)	118.4(4)	C(34)-C(22)-C(21)	122.1(4)
C(34)-C(22)-C(23)	119.4(4)	C(22)-C(23)-O(21)	124.8(4)
C(24)-C(23)-O(21)	113.9(4)	C(24)-C(23)-C(22)	121.3(4)
C(25)-C(24)-C(23)	114.5(3)	C(32)-C(24)-C(23)	112.8(3)
C(32)-C(24)-C(25)	102.6(3)	C(26)-C(25)-C(24)	115.0(3)
C(30)-C(25)-C(24)	102.6(4)	C(30)-C(25)-C(26)	112.5(3)
C(25)-C(26)-O(22)	114.3(4)	C(27)-C(26)-O(22)	124.0(4)
C(27)-C(26)-C(25)	121.6(4)	C(28)-C(27)-C(26)	117.2(4)
C(37)-C(27)-C(26)	118.9(4)	C(37)-C(27)-C(28)	123.9(4)
C(27)-C(28)-C(21)	116.8(3)	C(29)-C(28)-C(21)	89.4(3)
C(29)-C(28)-C(27)	116.0(3)	C(30)-C(29)-C(28)	118.1(3)
C(33)-C(29)-C(28)	90.8(3)	C(33)-C(29)-C(30)	103.5(3)
C(29)-C(30)-C(25)	108.8(3)	C(31)-C(30)-C(25)	101.8(3)
C(31)-C(30)-C(29)	101.3(3)	C(32)-C(31)-C(30)	94.8(3)
C(31)-C(32)-C(24)	101.7(3)	C(33)-C(32)-C(24)	108.5(3)
C(33)-C(32)-C(31)	100.9(3)	C(29)-C(33)-C(21)	90.7(3)
C(32)-C(33)-C(21)	118.0(3)	C(32)-C(33)-C(29)	103.6(3)
O(24)-C(34)-O(23)	122.2(4)	C(22)-C(34)-O(23)	113.7(4)
C(22)-C(34)-O(24)	124.1(4)	C(36)-C(35)-O(23)	107.6(5)
O(26)-C(37)-O(25)	121.6(4)	C(27)-C(37)-O(25)	114.3(4)
C(27)-C(37)-O(26)	124.1(5)	C(39)-C(38)-O(25)	107.4(4)

Abstract

2,4,6,8,10,12-Hexa(4-methoxybenzyl)-2,4,6,8,10,12-hexaazatetracyclo-[5.5.0.$3^{3,11}$.$0^{5,9}$]dodecane, $C_{54}H_{60}N_6O_6$, M_r = 889.11, monoclinic, $P2_1/c$, a = 16.419(2), b = 24.649(4), c = 12.358(2) Å, β = 109.73°, V = 4707.8(12) $Å^3$, Z = 4, D_x = 1.254 Mg m^{-3}, λ(Cu Kα) = 1.54178 R = 0.054, wR = 0.049, for 4162 independent reflections.

Experimental

A clear colorless 0.04 x 0.22 x 0.50 mm data crystal re-crystallized from octane/ N,N-dimethyl formamide mixed solvent was provided by A. Nielsen of NWC China Lake . Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within 50≤ 2θ ≤78° used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.55 $Å^{-1}$, range of hkl : -17 ≤ h ≤ 0, 0 ≤ k ≤ 26, 0 ≤≤ 13. Standards 220, 040, 102, monitored every 60 reflections with random variation of 2.5 % over data collection, θ/2θ mode, scan width $(2.0 + \Delta_{\alpha 1 \alpha 2})°$, scan rate a function of count rate (6.0°/min. minimum, 30°/min. maximum, 6465 reflections measured, 5793 unique, R_{int} = 0.01, 4162 observed with $Fo>3\sigma(F_o)$. Data corrected for Lorentz and polarization but not absorbtion effects . Secondary extinction parameter p = 0.0012(1) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. 614 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model , C-H = 0.96 Å, C-C-H = 109.5° , U(H) = 1.2 to 1.1 U_{eq}(C) . (Δ/σ) max = 0.24, R = 0.054, wR = 0.049, S = 1.39. Final difference Fourier excursions 0.18 and -0.20 e$Å^{-3}$. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1m).



2,4,6,8,10,12-Hexa(4-methoxybenzyl)-2,4,6,8,10,12-hexaazatetracyclo-[5.5.0.$3^{3,11}$.$0^{5,9}$]dodecane

Table 1m. Atom coordinates ($x10^4$) and thermal parameters ($Å^2 \times 10^3$)

atom	x	y	z	U
C(1)	1615(2)	6126(1)	3346(2)	41(1)*
N(2)	808(2)	6086(1)	2368(2)	37(1)*
C(3)	176(2)	6352(1)	2833(2)	38(1)*
N(4)	-321(2)	5959(1)	3220(2)	40(1)*
C(5)	237(2)	5622(1)	4147(2)	40(1)*
N(6)	911(2)	5297(1)	3959(2)	39(1)*
C(7)	1667(2)	5648(1)	4234(2)	41(1)*
N(8)	1711(2)	5881(1)	5351(2)	40(1)*
C(9)	780(2)	5996(1)	5160(2)	42(1)*
N(10)	557(2)	6556(1)	4892(2)	43(1)*
C(11)	705(2)	6724(1)	3827(2)	42(1)*
N(12)	1570(2)	6672(1)	3768(2)	39(1)*
C(13)	860(2)	6393(1)	1364(2)	51(1)*
C(14)	-919(2)	5659(1)	2249(3)	48(1)*
C(15)	730(2)	4881(1)	3074(3)	51(1)*
C(16)	2086(2)	5481(1)	6281(2)	52(1)*
C(17)	928(2)	6921(1)	5885(3)	53(1)*
C(18)	2315(2)	6936(1)	4567(3)	53(1)*
C(1a)	1534(2)	6176(1)	906(2)	45(1)*
C(2a)	1502(2)	5655(1)	485(3)	57(2)*
C(3a)	2141(2)	5450(2)	99(3)	60(2)*
C(4a)	2826(2)	5776(2)	125(3)	58(2)*
C(5a)	2859(2)	6304(2)	522(3)	66(2)*
C(6a)	2218(2)	6494(1)	912(3)	57(2)*
O(7a)	3511(2)	5622(1)	-205(2)	89(1)*
C(8a)	3593(3)	5069(2)	-425(4)	85(2)*
C(1b)	-1745(2)	5962(1)	1656(3)	42(1)*
C(2b)	-2333(2)	6059(1)	2208(3)	55(2)*
C(3b)	-3141(2)	6277(1)	1652(3)	59(2)*
C(4b)	-3377(2)	6405(1)	497(3)	54(2)*
C(5b)	-2794(2)	6336(2)	-54(3)	62(2)*
C(6b)	-1988(2)	6118(1)	510(3)	55(2)*
O(7b)	-4171(2)	6603(1)	-142(2)	78(1)*
C(8b)	-4870(3)	6516(2)	234(4)	102(2)*
C(1c)	1438(2)	4460(1)	3379(3)	44(1)*
C(2c)	1498(2)	4075(1)	4214(3)	46(1)*
C(3c)	2145(2)	3692(1)	4516(3)	47(1)*
C(4c)	2759(2)	3689(1)	3989(3)	53(2)*
C(5c)	2726(2)	4074(1)	3170(3)	65(2)*
C(6c)	2066(2)	4449(1)	2861(3)	59(2)*
O(7c)	3434(2)	3325(1)	4216(2)	78(1)*
C(8c)	3446(3)	2893(2)	4967(4)	86(2)*
C(1d)	3037(2)	5393(1)	6520(3)	44(1)*
C(2d)	3651(3)	5766(2)	7105(3)	60(2)*
C(3d)	4517(3)	5680(2)	7325(3)	70(2)*
C(4d)	4800(3)	5208(2)	6961(3)	60(2)*
C(5d)	4205(3)	4834(2)	6368(3)	61(2)*
C(6d)	3333(2)	4929(1)	6158(3)	56(2)*
O(7d)	5680(2)	5160(1)	7240(2)	88(1)*
C(8d)	5998(3)	4675(2)	6906(4)	105(3)*
C(1e)	360(2)	6955(1)	6619(2)	44(1)*
C(2e)	-340(2)	7295(1)	6309(3)	47(1)*
C(3e)	-890(2)	7341(1)	6941(3)	49(1)*
C(4e)	-733(3)	7026(2)	7915(3)	55(2)*
C(5e)	-37(3)	6680(2)	8236(3)	65(2)*
C(6e)	512(2)	6647(1)	7603(3)	62(2)*
O(7e)	-1228(2)	7031(1)	8618(2)	79(1)*
C(8e)	-1901(3)	7416(2)	8366(4)	103(3)*
C(1f)	3020(2)	7033(1)	4061(3)	47(1)*
C(2f)	2978(2)	7471(1)	3354(3)	58(2)*
C(3f)	3606(3)	7560(2)	2849(3)	64(2)*
C(4f)	4277(2)	7203(2)	3049(3)	59(2)*
C(5f)	4337(2)	6767(2)	3763(3)	65(2)*
C(6f)	3710(2)	6682(1)	4256(3)	57(2)*
O(7f)	4938(2)	7245(1)	2599(2)	83(1)*
C(8f)	5023(3)	7739(2)	2067(4)	115(3)*

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2m. Bond lengths (Å)

C(1)-N(2)	1.465(3)	C(1)-C(7)	1.592(4)
C(1)-N(12)	1.454(4)	N(2)-C(3)	1.496(4)
N(2)-C(13)	1.481(4)	C(3)-N(4)	1.448(4)
C(3)-C(11)	1.544(4)	N(4)-C(5)	1.461(3)
N(4)-C(14)	1.469(3)	C(5)-N(6)	1.447(4)
C(5)-C(9)	1.568(4)	N(6)-C(7)	1.456(4)
N(6)-C(15)	1.456(4)	C(7)-N(8)	1.475(4)
N(8)-C(9)	1.492(4)	N(8)-C(16)	1.481(4)
C(9)-N(10)	1.438(4)	N(10)-C(11)	1.476(4)
N(10)-C(17)	1.478(4)	C(11)-N(12)	1.452(4)
N(12)-C(18)	1.443(3)	C(13)-C(1a)	1.500(5)
C(14)-C(1b)	1.504(4)	C(15)-C(1c)	1.508(4)
C(16)-C(1d)	1.503(5)	C(17)-C(1e)	1.506(5)
C(18)-C(1f)	1.508(5)	C(1a)-C(2a)	1.380(5)
C(1a)-C(6a)	1.369(5)	C(2a)-C(3a)	1.385(6)
C(3a)-C(4a)	1.374(6)	C(4a)-C(5a)	1.384(5)
C(4a)-O(7a)	1.373(5)	C(5a)-C(6a)	1.380(6)
O(7a)-C(8a)	1.405(5)	C(1b)-C(2b)	1.378(6)
C(1b)-C(6b)	1.390(4)	C(2b)-C(3b)	1.381(5)
C(3b)-C(4b)	1.384(5)	C(4b)-C(5b)	1.359(6)
C(4b)-O(7b)	1.366(4)	C(5b)-C(6b)	1.380(5)
O(7b)-C(8b)	1.394(6)	C(1c)-C(2c)	1.381(4)
C(1c)-C(6c)	1.385(6)	C(2c)-C(3c)	1.375(4)
C(3c)-C(4c)	1.372(6)	C(4c)-C(5c)	1.374(5)
C(4c)-O(7c)	1.379(4)	C(5c)-C(6c)	1.377(5)
O(7c)-C(8c)	1.408(5)	C(1d)-C(2d)	1.377(5)
C(1d)-C(6d)	1.375(5)	C(2d)-C(3d)	1.371(6)
C(3d)-C(4d)	1.382(6)	C(4d)-C(5d)	1.364(5)
C(4d)-O(7d)	1.373(5)	C(5d)-C(6d)	1.386(6)
O(7d)-C(8d)	1.419(6)	C(1e)-C(2e)	1.368(4)
C(1e)-C(6e)	1.383(4)	C(2e)-C(3e)	1.384(5)
C(3e)-C(4e)	1.382(5)	C(4e)-C(5e)	1.373(5)
C(4e)-O(7e)	1.376(5)	C(5e)-C(6e)	1.379(6)
O(7e)-C(8e)	1.409(5)	C(1f)-C(2f)	1.377(5)
C(1f)-C(6f)	1.380(5)	C(2f)-C(3f)	1.391(6)
C(3f)-C(4f)	1.366(6)	C(4f)-C(5f)	1.373(5)
C(4f)-O(7f)	1.380(5)	C(5f)-C(6f)	1.378(6)
O(7f)-C(8f)	1.414(6)		

Table 3m. Bond angles (deg.)

N(2)-C(1)-C(7)	110.0(2)	N(2)-C(1)-N(12)	102.5(2)
C(7)-C(1)-N(12)	115.8(2)	C(1)-N(2)-C(3)	101.9(2)
C(1)-N(2)-C(13)	111.3(2)	C(3)-N(2)-C(13)	110.0(2)
N(2)-C(3)-N(4)	112.0(2)	N(2)-C(3)-C(11)	106.8(2)
N(4)-C(3)-C(11)	111.2(2)	C(3)-N(4)-C(5)	111.4(2)
C(3)-N(4)-C(14)	111.3(2)	C(5)-N(4)-C(14)	115.0(2)
N(4)-C(5)-N(6)	120.0(3)	N(4)-C(5)-C(9)	109.2(2)
N(6)-C(5)-C(9)	101.1(2)	C(5)-N(6)-C(7)	105.8(2)
C(5)-N(6)-C(15)	122.5(2)	C(7)-N(6)-C(15)	122.4(3)
C(1)-C(7)-N(6)	115.8(2)	C(1)-C(7)-N(8)	109.4(2)
N(6)-C(7)-N(8)	102.5(3)	C(7)-N(8)-C(9)	101.7(2)
C(7)-N(8)-C(16)	110.1(2)	C(9)-N(8)-C(16)	112.6(2)
C(5)-C(9)-N(8)	106.9(2)	C(5)-C(9)-N(10)	110.4(2)
N(8)-C(9)-N(10)	112.7(3)	C(9)-N(10)-C(11)	111.6(3)
C(9)-N(10)-C(17)	113.1(2)	C(11)-N(10)-C(17)	115.1(2)
C(3)-C(11)-N(10)	109.0(2)	C(3)-C(11)-N(12)	101.8(2)
N(10)-C(11)-N(12)	118.6(2)	C(1)-N(12)-C(11)	105.6(2)
C(1)-N(12)-C(18)	122.0(2)	C(11)-N(12)-C(18)	122.9(3)
N(2)-C(13)-C(1a)	113.1(3)	N(4)-C(14)-C(1b)	113.0(3)
N(6)-C(15)-C(1c)	110.6(2)	N(8)-C(16)-C(1d)	112.4(3)
N(10)-C(17)-C(1e)	111.6(3)	N(12)-C(18)-C(1f)	112.3(3)
C(13)-C(1a)-C(2a)	122.4(3)	C(13)-C(1a)-C(6a)	120.1(3)
C(2a)-C(1a)-C(6a)	117.5(4)	C(1a)-C(2a)-C(3a)	122.1(3)
C(2a)-C(3a)-C(4a)	119.3(3)	C(3a)-C(4a)-C(5a)	119.4(4)
C(3a)-C(4a)-O(7a)	125.3(3)	C(5a)-C(4a)-O(7a)	115.3(3)
C(4a)-C(5a)-C(6a)	120.0(4)	C(1a)-C(6a)-C(5a)	121.6(3)
C(4a)-O(7a)-C(8a)	117.9(3)	C(14)-C(1b)-C(2b)	120.8(3)
C(14)-C(1b)-C(6b)	122.3(3)	C(2b)-C(1b)-C(6b)	116.6(3)
C(1b)-C(2b)-C(3b)	122.6(3)	C(2b)-C(3b)-C(4b)	119.2(4)
C(3b)-C(4b)-C(5b)	119.4(3)	C(3b)-C(4b)-O(7b)	123.8(4)
C(5b)-C(4b)-O(7b)	116.8(3)	C(4b)-C(5b)-C(6b)	120.9(3)
C(1b)-C(6b)-C(5b)	121.2(4)	C(4b)-O(7b)-C(8b)	119.1(3)
C(15)-C(1c)-C(2c)	120.9(3)	C(15)-C(1c)-C(6c)	122.0(3)
C(2c)-C(1c)-C(6c)	117.1(3)	C(1c)-C(2c)-C(3c)	121.8(3)
C(2c)-C(3c)-C(4c)	120.0(3)	C(3c)-C(4c)-C(5c)	119.6(3)
C(3c)-C(4c)-O(7c)	125.1(3)	C(5c)-C(4c)-O(7c)	115.3(4)
C(4c)-C(5c)-C(6c)	119.9(4)	C(1c)-C(6c)-C(5c)	121.6(3)
C(4c)-O(7c)-C(8c)	117.3(3)	C(16)-C(1d)-C(2d)	122.2(3)
C(16)-C(1d)-C(6d)	121.0(3)	C(2d)-C(1d)-C(6d)	116.8(3)
C(1d)-C(2d)-C(3d)	121.8(4)	C(2d)-C(3d)-C(4d)	120.4(3)
C(3d)-C(4d)-C(5d)	119.0(4)	C(3d)-C(4d)-O(7d)	115.7(3)
C(5d)-C(4d)-O(7d)	125.3(4)	C(4d)-C(5d)-C(6d)	119.7(4)
C(1d)-C(6d)-C(5d)	122.3(3)	C(4d)-O(7d)-C(8d)	117.4(3)
C(17)-C(1e)-C(2e)	119.9(3)	C(17)-C(1e)-C(6e)	122.4(3)
C(2e)-C(1e)-C(6e)	117.8(3)	C(1e)-C(2e)-C(3e)	122.7(3)
C(2e)-C(3e)-C(4e)	118.6(3)	C(3e)-C(4e)-C(5e)	119.6(4)
C(3e)-C(4e)-O(7e)	124.6(3)	C(5e)-C(4e)-O(7e)	115.8(3)
C(4e)-C(5e)-C(6e)	120.7(3)	C(1e)-C(6e)-C(5e)	120.6(3)
C(4e)-O(7e)-C(8e)	116.9(3)	C(18)-C(1f)-C(2f)	120.4(3)
C(18)-C(1f)-C(6f)	121.9(3)	C(2f)-C(1f)-C(6f)	117.7(3)
C(1f)-C(2f)-C(3f)	121.6(3)	C(2f)-C(3f)-C(4f)	119.4(4)
C(3f)-C(4f)-C(5f)	120.0(4)	C(3f)-C(4f)-O(7f)	125.1(4)
C(5f)-C(4f)-O(7f)	114.9(3)	C(4f)-C(5f)-C(6f)	120.1(3)
C(1f)-C(6f)-C(5f)	121.2(3)	C(4f)-O(7f)-C(8f)	117.8(3)

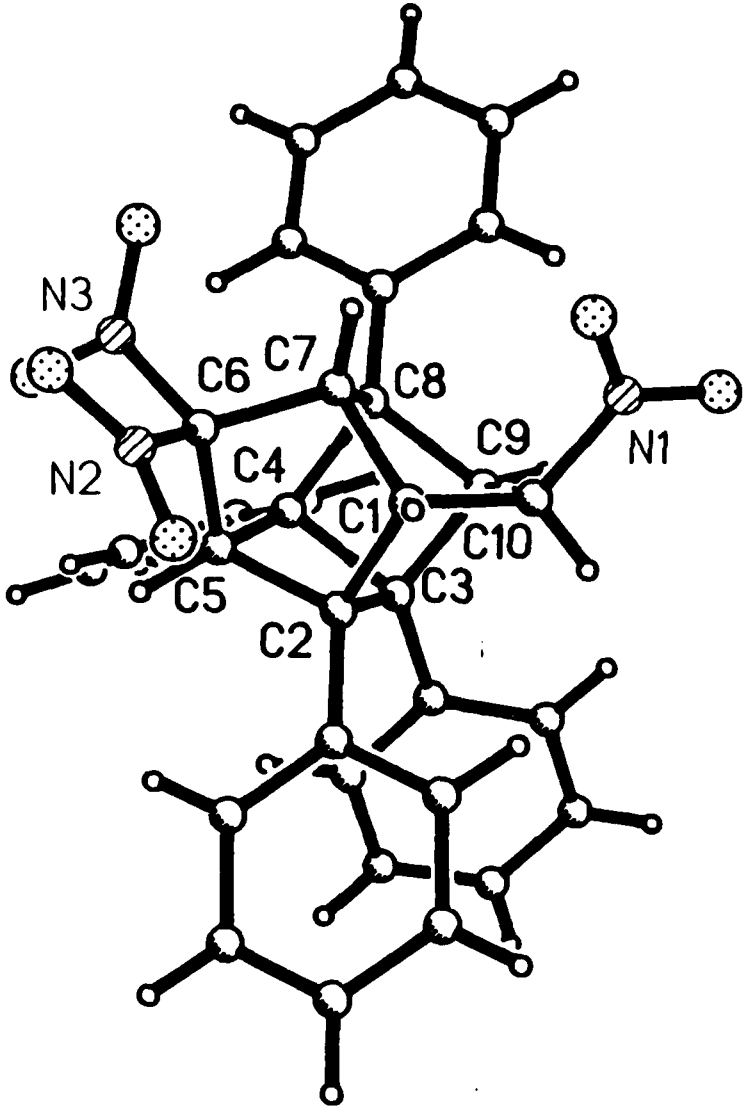
Abstract

2,3,4,8-Tetraphenyl-6,6-anti-10-trinitropentacyclo[5.3.0.$0^{2,5}$.$0^{3,9}$.$0^{4,8}$]decane, $C_{34}H_{25}N_3O_6$, M_r = 571.59, monoclinic, $P2_1/c$, a = 10.869(4), b = 15.667(7), c = 17.084(8) Å, β = 108.36(4)°, V = 2760.9(23) Å^3, Z = 4, D_x = 1.37 Mg m^{-3}, λ(Mo Kα) = 0.71069 Å, μ = 0.90 cm^{-1}, F(000) = 1192, T = 295 K, Final R = 0.052, wR = 0.054 for 3568 independent reflections.

Experimental

A clear colorless 0.25 x 0.50 x 0.09 mm data crystal recrystallized from ethyl acetate hexane mixed solvent was provided by A. Marchand of North Texas State University. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within $30 \leq 2\theta \leq 35°$ used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.59 Å^{-1}, range of hkl : $-12 \leq h \leq 11$, $0 \leq k \leq 18$, $0 \leq l \leq 18$. Standards 600, 0 10 0, 0 0 10, monitored every 60 reflections with random variation of 2.0 % over data collection, θ/2θ mode, scan width [2θ(Kα$_1$) - 0.7 to [2θ(Kα$_2$) + 0.7]°, scan rate a function of count rate (12.0°/min. minimum, 30.0°/min. maximum, 5538 reflections measured, 4899 unique, R_{int} = 0.013, 3568 observed with $Fo > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorbtion effects . Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. 388 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model (coordinate shifts of C applied to attached H atoms), C-H = 0.96 Å, U(H) = 1.1 U_{eq}(C) . (Δ/σ) max = 0.12, R = 0.052, wR = 0.054. Final difference Fourier excursions 0.23 and -0.23 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, and 2, atom coordinates, bond distances and angles, follows that shown in Fig.(1n).

2,3,4,8-Tetraphenyl-6,6-anti-10-trinitropentacyclo[5.3.0.0^{2,5}.0^{3,9}.0^{4,8}]decane



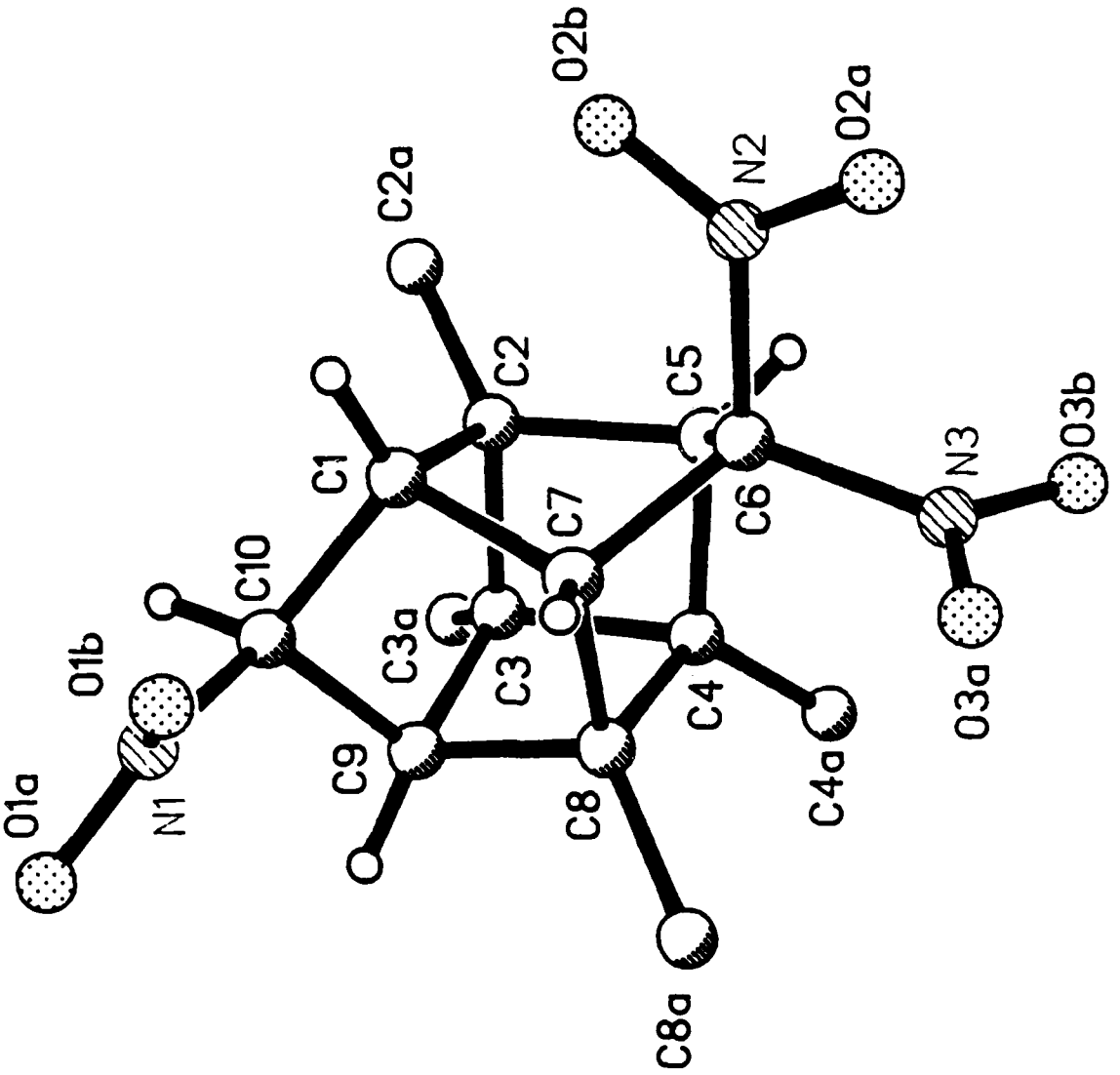

O2b
O2a
N2
C2a
C2
C5
C1
C7
C6
N3
O3b
C10
C3a
C3
C4
O3a
O1b
O1a
N1
C9
C8
C4a
C8a

Table 1n. Atomic coordinates and equivalent isotropic displacement parameters ($A^2 x 10^2$)

	x	y	z	U(eq)
N(1)	0.1204(2)	0.0575(1)	0.1082(1)	4.8(1)
O(1A)	0.1427(2)	0.0484(1)	0.0433(1)	7.9(1)
O(1B)	0.1968(2)	0.0386(1)	0.1749(1)	7.8(1)
N(2)	-.0274(2)	0.1727(1)	0.3526(1)	4.8(1)
O(2A)	0.0321(2)	0.1941(1)	0.4218(1)	7.5(1)
O(2B)	-.0976(2)	0.1114(1)	0.3348(1)	8.1(1)
N(3)	0.0570(2)	0.3054(1)	0.3253(1)	5.0(1)
O(3A)	0.1752(2)	0.3046(1)	0.3501(1)	7.1(1)
O(3B)	-.0107(2)	0.3649(1)	0.3330(1)	7.0(1)
C(1)	-.0316(2)	0.1101(1)	0.1858(1)	3.8(1)
C(2)	-.1651(2)	0.1590(1)	0.1599(1)	3.5(1)
C(3)	-.1561(2)	0.2139(1)	0.0820(1)	3.3(1)
C(4)	-.0992(2)	0.2882(1)	0.1451(1)	3.3(1)
C(5)	-.1373(2)	0.2423(1)	0.2140(1)	3.4(1)
C(6)	-.0110(2)	0.2263(1)	0.2815(1)	3.8(1)
C(7)	0.0600(2)	0.1830(1)	0.2286(1)	3.6(1)
C(8)	0.0407(2)	0.2476(1)	0.1539(1)	3.3(1)
C(9)	-.0219(2)	0.1900(1)	0.0767(1)	3.5(1)
C(10)	-.0067(2)	0.0975(2)	0.1040(1)	4.0(1)
C(2A)	-.2852(2)	0.1073(1)	0.1512(1)	3.9(1)
C(2B)	-.3832(2)	0.1367(2)	0.1789(2)	6.0(1)
C(2C)	-.4967(2)	0.0902(2)	0.1665(2)	7.3(1)
C(2D)	-.5130(3)	0.0146(2)	0.1266(2)	6.5(1)
C(2E)	-.4158(3)	-.0171(2)	0.0999(2)	6.5(1)
C(2F)	-.3028(3)	0.0290(2)	0.1121(2)	5.4(1)
C(3A)	-.2703(2)	0.2224(1)	0.0057(1)	3.7(1)
C(3B)	-.2694(2)	0.1872(2)	-.0686(1)	4.7(1)
C(3C)	-.3747(3)	0.1952(2)	-.1389(2)	5.9(1)
C(3D)	-.4832(3)	0.2382(2)	-.1365(2)	6.8(1)
C(3E)	-.4866(3)	0.2736(2)	-.0640(2)	7.0(1)
C(3F)	-.3811(2)	0.2660(2)	0.0068(2)	5.4(1)
C(4A)	-.1363(2)	0.3786(1)	0.1204(1)	3.5(1)
C(4B)	-.2053(2)	0.4280(2)	0.1599(2)	4.6(1)
C(4C)	-.2535(3)	0.5074(2)	0.1286(2)	5.7(1)
C(4D)	-.2319(3)	0.5383(2)	0.0589(2)	6.3(1)
C(4E)	-.1617(3)	0.4909(2)	0.0204(2)	5.9(1)
C(4F)	-.1148(2)	0.4119(2)	0.0505(1)	4.5(1)
C(8A)	0.1572(2)	0.3010(2)	0.1552(1)	3.6(1)
C(8B)	0.2485(2)	0.2667(2)	0.1225(1)	4.6(1)
C(8C)	0.3590(2)	0.3125(2)	0.1249(2)	5.6(1)
C(8D)	0.3794(2)	0.3921(2)	0.1584(2)	5.8(1)
C(8E)	0.2890(2)	0.4277(2)	0.1898(2)	5.3(1)
C(8F)	0.1791(2)	0.3823(2)	0.1885(1)	4.4(1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2n. Bond lengths (Å)

N(1)-O(1A)	1.215	(3)	N(1)-O(1B)	1.216	(2)
N(1)-C(10)	1.498	(3)	N(2)-O(2A)	1.204	(2)
N(2)-O(2B)	1.205	(3)	N(2)-C(6)	1.532	(3)
N(3)-O(3A)	1.219	(2)	N(3)-O(3B)	1.221	(3)
N(3)-C(6)	1.513	(3)	C(1)-C(2)	1.575	(3)
C(1)-C(7)	1.540	(3)	C(1)-C(10)	1.518	(3)
C(2)-C(3)	1.614	(3)	C(2)-C(5)	1.573	(3)
C(2)-C(2A)	1.503	(3)	C(3)-C(4)	1.575	(3)
C(3)-C(9)	1.536	(3)	C(3)-C(3A)	1.496	(3)
C(4)-C(5)	1.543	(3)	C(4)-C(8)	1.611	(3)
C(4)-C(4A)	1.496	(3)	C(5)-C(6)	1.510	(3)
C(6)-C(7)	1.521	(3)	C(7)-C(8)	1.590	(3)
C(8)-C(9)	1.565	(3)	C(8)-C(8A)	1.512	(3)
C(9)-C(10)	1.516	(3)	C(2A)-C(2B)	1.374	(3)
C(2A)-C(2F)	1.381	(3)	C(2B)-C(2C)	1.391	(3)
C(2C)-C(2D)	1.351	(4)	C(2D)-C(2E)	1.368	(4)
C(2E)-C(2F)	1.383	(3)	C(3A)-C(3B)	1.387	(3)
C(3A)-C(3F)	1.389	(3)	C(3B)-C(3C)	1.379	(3)
C(3C)-C(3D)	1.370	(4)	C(3D)-C(3E)	1.368	(4)
C(3E)-C(3F)	1.385	(3)	C(4A)-C(4B)	1.390	(3)
C(4A)-C(4F)	1.389	(3)	C(4B)-C(4C)	1.389	(3)
C(4C)-C(4D)	1.372	(4)	C(4D)-C(4E)	1.373	(4)
C(4E)-C(4F)	1.375	(3)	C(8A)-C(8B)	1.390	(3)
C(8A)-C(8F)	1.384	(3)	C(8B)-C(8C)	1.389	(3)
C(8C)-C(8D)	1.360	(4)	C(8D)-C(8E)	1.378	(4)
C(8E)-C(8F)	1.384	(3)			

Bond angles (°)

O(1B)-N(1)-O(1A)	123.6(2)	C(10)-N(1)-O(1A)	116.9(2)
C(10)-N(1)-O(1B)	119.5(2)	O(2B)-N(2)-O(2A)	124.9(2)
C(6)-N(2)-O(2A)	117.7(2)	C(6)-N(2)-O(2B)	117.4(2)
O(3B)-N(3)-O(3A)	125.2(2)	C(6)-N(3)-O(3A)	117.4(2)
C(6)-N(3)-O(3B)	117.4(2)	C(7)-C(1)-C(2)	100.0(2)
C(10)-C(1)-C(2)	102.7(2)	C(10)-C(1)-C(7)	104.9(2)
C(3)-C(2)-C(1)	101.6(2)	C(5)-C(2)-C(1)	104.4(2)
C(5)-C(2)-C(3)	89.5(1)	C(2A)-C(2)-C(1)	117.4(2)
C(2A)-C(2)-C(3)	118.8(2)	C(2A)-C(2)-C(5)	120.4(2)
C(4)-C(3)-C(2)	86.7(1)	C(9)-C(3)-C(2)	102.9(2)
C(9)-C(3)-C(4)	92.6(2)	C(3A)-C(3)-C(2)	121.2(2)
C(3A)-C(3)-C(4)	124.7(2)	C(3A)-C(3)-C(9)	120.9(2)
C(5)-C(4)-C(3)	92.0(2)	C(8)-C(4)-C(3)	85.8(1)
C(8)-C(4)-C(5)	102.5(2)	C(4A)-C(4)-C(3)	119.7(2)
C(4A)-C(4)-C(5)	122.9(2)	C(4A)-C(4)-C(8)	124.3(2)
C(4)-C(5)-C(2)	89.3(2)	C(6)-C(5)-C(2)	105.7(2)
C(6)-C(5)-C(4)	105.3(2)	N(3)-C(6)-N(2)	102.6(2)
C(5)-C(6)-N(2)	113.1(2)	C(5)-C(6)-N(3)	115.3(2)
C(7)-C(6)-N(2)	115.7(2)	C(7)-C(6)-N(3)	113.5(2)
C(7)-C(6)-C(5)	97.4(2)	C(6)-C(7)-C(1)	103.9(2)
C(8)-C(7)-C(1)	100.9(2)	C(8)-C(7)-C(6)	103.3(2)
C(7)-C(8)-C(4)	102.0(2)	C(9)-C(8)-C(4)	90.1(1)
C(9)-C(8)-C(7)	102.8(2)	C(8A)-C(8)-C(4)	123.0(2)
C(8A)-C(8)-C(7)	116.2(2)	C(8A)-C(8)-C(9)	118.1(2)
C(8)-C(9)-C(3)	88.8(2)	C(10)-C(9)-C(3)	103.2(2)
C(10)-C(9)-C(8)	108.4(2)	C(1)-C(10)-N(1)	116.1(2)
C(9)-C(10)-N(1)	115.0(2)	C(9)-C(10)-C(1)	97.2(2)
C(2B)-C(2A)-C(2)	121.9(2)	C(2F)-C(2A)-C(2)	120.7(2)
C(2F)-C(2A)-C(2B)	117.4(2)	C(2C)-C(2B)-C(2A)	121.3(3)
C(2D)-C(2C)-C(2B)	120.3(3)	C(2E)-C(2D)-C(2C)	119.6(2)

C(2F)-C(2E)-C(2D)	120.1(3)	C(2E)-C(2F)-C(2A)	121.3(2)
C(3B)-C(3A)-C(3)	121.2(2)	C(3F)-C(3A)-C(3)	121.1(2)
C(3F)-C(3A)-C(3B)	117.6(2)	C(3C)-C(3B)-C(3A)	121.1(2)
C(3D)-C(3C)-C(3B)	120.3(3)	C(3E)-C(3D)-C(3C)	119.7(3)
C(3F)-C(3E)-C(3D)	120.3(3)	C(3E)-C(3F)-C(3A)	120.9(2)
C(4B)-C(4A)-C(4)	121.9(2)	C(4F)-C(4A)-C(4)	119.6(2)
C(4F)-C(4A)-C(4B)	118.1(2)	C(4C)-C(4B)-C(4A)	120.5(2)
C(4D)-C(4C)-C(4B)	120.2(2)	C(4E)-C(4D)-C(4C)	119.8(3)
C(4F)-C(4E)-C(4D)	120.3(2)	C(4E)-C(4F)-C(4A)	121.1(2)
C(8B)-C(8A)-C(8)	118.7(2)	C(8F)-C(8A)-C(8)	123.3(2)
C(8F)-C(8A)-C(8B)	117.9(2)	C(8C)-C(8B)-C(8A)	120.6(2)
C(8D)-C(8C)-C(8B)	120.7(2)	C(8E)-C(8D)-C(8C)	119.6(2)
C(8F)-C(8E)-C(8D)	120.2(2)	C(8E)-C(8F)-C(8A)	121.0(2)

Abstract

1,6-Dimethyl-1α-4α,4aα,5α 8β,8aα-hexahydro-1,4-methanonaphthalene, $C_{13}H_{18}O_2$, M_r = 206.26, monoclinic, $P2_1/c$, a = 9.830(2), b = 9.618(3), c = 12.584(4) Å, β = 111.29(2)°, V = 1108.5(6) Å^3, Z = 4, D_x = 1.24 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 6.10 cm^{-1}, F(000) = 448, T = 295 K, Final R = 0.078, wR = 0.088 for 1252 independent reflections. The hydroxyl groups are both on the same side of the six-membered ring.

Experimental

A clear colorless data crystal grown from acetone was provided by A. Marchand of North Texas State University. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within $50 \leq 2\theta \leq 80°$ used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.55 Å^{-1}, range of hkl : $-10 \leq h \leq 0$, $0 \leq k \leq 10$, $-12 \leq l \leq 12$. Standards 204, 040, 302, monitored every 60 reflections with linear variation of 2.9 % over data collection, θ/2θ mode, scan width [2θ(Kα$_1$) - 1.0] to [2θ(Kα$_2$) + 1.0]°, scan rate a function of count rate (14.0°/min. minimum, 30.0°/min. maximum), 1846 reflections measured, 1524 unique, R_{int} = 0.022, 1252 observed with $Fo > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorbtion effects . Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. 149 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model (coordinate shifts of C applied to attached H atoms), C-H = 0.96 Å, C-C-H = 109.5° (methyl H) , $U(H) = 1.2\ U_{eq}(C)$. R = 0.078, wR = 0.088. Final difference Fourier excursions 0.31 and -0.35 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, and 2, atom coordinates, bond distances and angles, follows that shown in Fig.(1o).

1,6-Dimethyl-1α-4α,4aα,5α 8β,8aα-hexahydro-1,4-methanonaphthalene

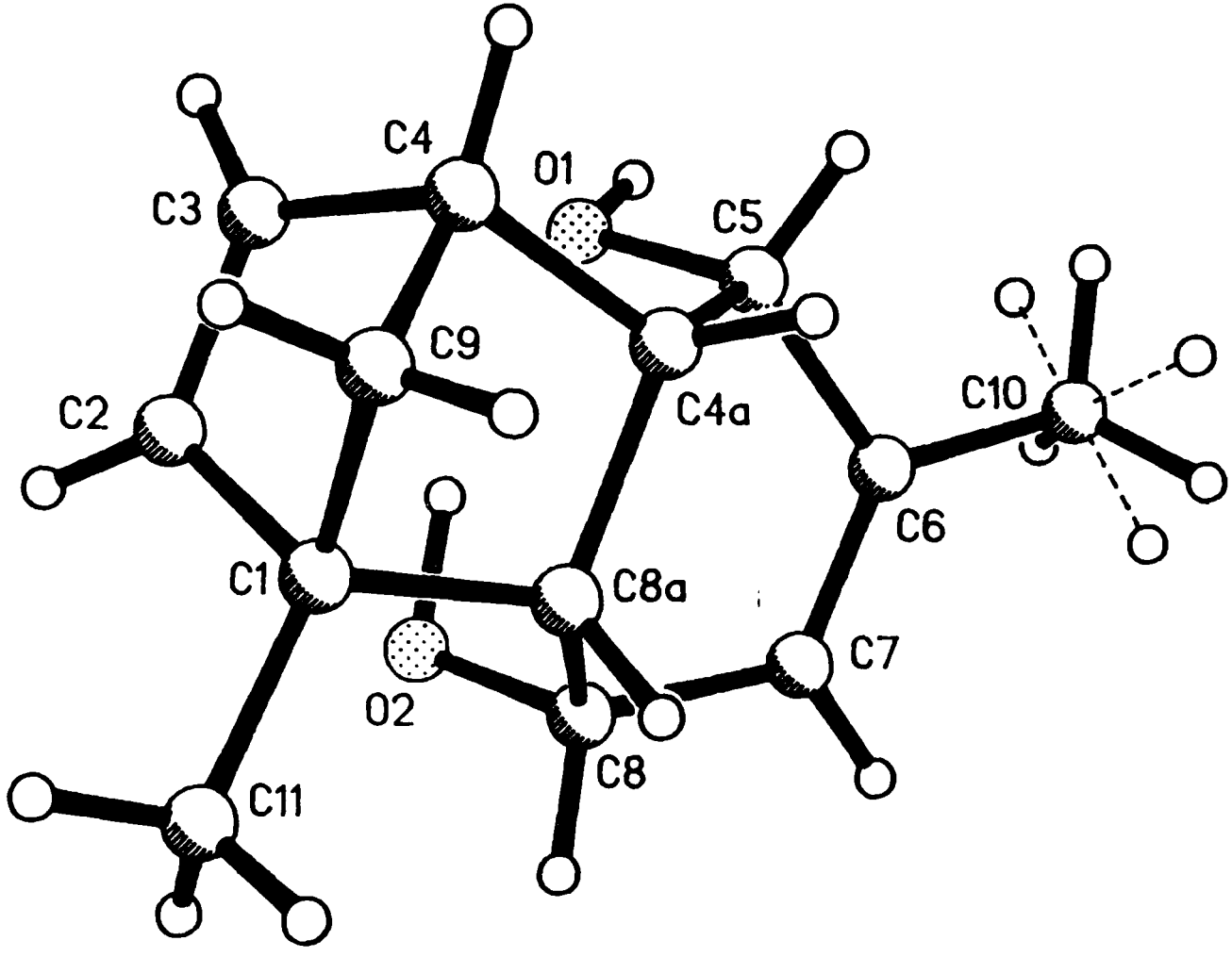


Table 1o. Atomic coordinates and equivalent isotropic displacement parameters ($A^2 x 10^2$)

	x	y	z	U(eq)
O(1)	0.6022(4)	0.1169(3)	0.2217(3)	4.9(1)
O(2)	0.5280(3)	-.1197(3)	0.3224(3)	4.9(1)
C(1)	0.7979(5)	-.2462(4)	0.2907(4)	4.7(2)
C(2)	0.6724(5)	-.2215(4)	0.1804(4)	4.7(2)
C(3)	0.7017(5)	-.1079(4)	0.1318(4)	4.9(2)
C(4)	0.8484(5)	-.0561(4)	0.2085(4)	5.1(2)
C(4a)	0.8368(5)	0.0045(4)	0.3187(3)	4.4(2)
C(5)	0.7401(5)	0.1305(4)	0.3136(3)	4.1(2)
C(6)	0.7171(5)	0.1440(4)	0.4264(4)	4.6(2)
C(7)	0.6829(5)	0.0308(4)	0.4712(4)	5.1(2)
C(8)	0.6684(5)	-.1090(4)	0.4129(4)	4.6(2)
C(8a)	0.7988(5)	-.1274(4)	0.3744(4)	4.4(2)
C(9)	0.9232(5)	-.1936(4)	0.2538(5)	5.9(2)
C(10)	0.7229(6)	0.2883(5)	0.4746(4)	5.9(2)
C(11)	0.8087(6)	-.3916(4)	0.3405(5)	6.5(2)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2o. Bond lengths (A)

O(1)-C(5)	1.433 (5)	O(2)-C(8)	1.438 (5)
C(1)-C(2)	1.504 (6)	C(1)-C(8a)	1.552 (6)
C(1)-C(9)	1.550 (6)	C(1)-C(11)	1.520 (6)
C(2)-C(3)	1.334 (6)	C(3)-C(4)	1.499 (6)
C(4)-C(4a)	1.547 (6)	C(4)-C(9)	1.519 (6)
C(4a)-C(5)	1.526 (5)	C(4a)-C(8a)	1.559 (5)
C(5)-C(6)	1.521 (6)	C(6)-C(7)	1.323 (6)
C(6)-C(10)	1.507 (6)	C(7)-C(8)	1.514 (5)
C(8)-C(8a)	1.535 (6)		

Bond angles (^{o})

C(8a)-C(1)-C(2)	108.1(3)	C(9)-C(1)-C(2)	98.4(4)
C(9)-C(1)-C(8a)	98.8(3)	C(11)-C(1)-C(2)	115.9(4)
C(11)-C(1)-C(8a)	114.6(4)	C(11)-C(1)-C(9)	118.7(4)
C(3)-C(2)-C(1)	108.2(4)	C(4)-C(3)-C(2)	107.1(4)
C(4a)-C(4)-C(3)	109.6(4)	C(9)-C(4)-C(3)	100.0(4)
C(9)-C(4)-C(4a)	100.1(3)	C(5)-C(4a)-C(4)	120.6(4)
C(8a)-C(4a)-C(4)	101.7(3)	C(8a)-C(4a)-C(5)	114.7(3)
C(4a)-C(5)-O(1)	110.7(3)	C(6)-C(5)-O(1)	110.1(4)
C(6)-C(5)-C(4a)	109.2(3)	C(7)-C(6)-C(5)	118.2(4)
C(10)-C(6)-C(5)	117.2(4)	C(10)-C(6)-C(7)	124.4(4)
C(8)-C(7)-C(6)	121.2(4)	C(7)-C(8)-O(2)	110.0(4)
C(8a)-C(8)-O(2)	114.4(3)	C(8a)-C(8)-C(7)	108.2(4)
C(4a)-C(8a)-C(1)	103.8(3)	C(8)-C(8a)-C(1)	119.9(4)
C(8)-C(8a)-C(4a)	114.3(3)	C(4)-C(9)-C(1)	93.9(3)

Abstract

2,2-Bis(trifluoromethyl)-4,5-diacetoxy-1,3-diacetyl-imidazolidine, $C_{13}H_{14}N_2O_6F_6$, M_r = 408.25, monoclinic , $P2_1/n$, a = 10.068(2), b = 22.076(4), c = 16.071(30 Å, β = 105.68(2)°, V = 3439.2(9) Å^3, Z = 8, D_x = 1.578 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 13.17 cm^{-1}, F(000) = 1632, T = 295 K, Final R = 0.072, wR = 0.080 for 4032 independent reflections. There are two molecules in the asymmetric unit.

Experimental

A clear colorless 0.25 x 0.12 x 0.15 mm data crystal recrystallized from ethylene dichloride was provided by W. Koppes of NSWC White Oak. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within $50 \leq 2\theta \leq 75°$ used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.58 Å^{-1}, range of hkl : $0 \leq h \leq 11$, $0 \leq k \leq 25$, $-17 \leq l \leq 17$. Standards 400, 080, 004, monitored every 60 reflections with random variation of 2.3 % over data collection, $\theta/2\theta$ mode, scan width [$2\theta(K\alpha_1)$ - 1.0] to [$2\theta(K\alpha_2)$ + 1.0]°, scan rate a function of count rate (12°/min. minimum, 30°/min. maximum, 6325 reflections measured, 5495 unique, R_{int} = 0.008, 4032 observed with $Fo > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorbtion effects . Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. Secondary extinction parameter p = 0.0007(2) in $F_c^* = F_c/[\ 1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. 512 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms,H atoms included using riding model , C-H = 0.96 Å, C-C-H = 109.5° , $U(H) = 1.1 \cdot U_{eq}(C)$. (Δ/σ) max = 0.23, R = 0.072, wR = 0.080, S = 2.382. Final difference Fourier excursions 0.33 and -0.36 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1p).

2,2-Bis(trifluoromethyl)-4,5-diacetoxy-1,3-diacetyl-imidazolidine

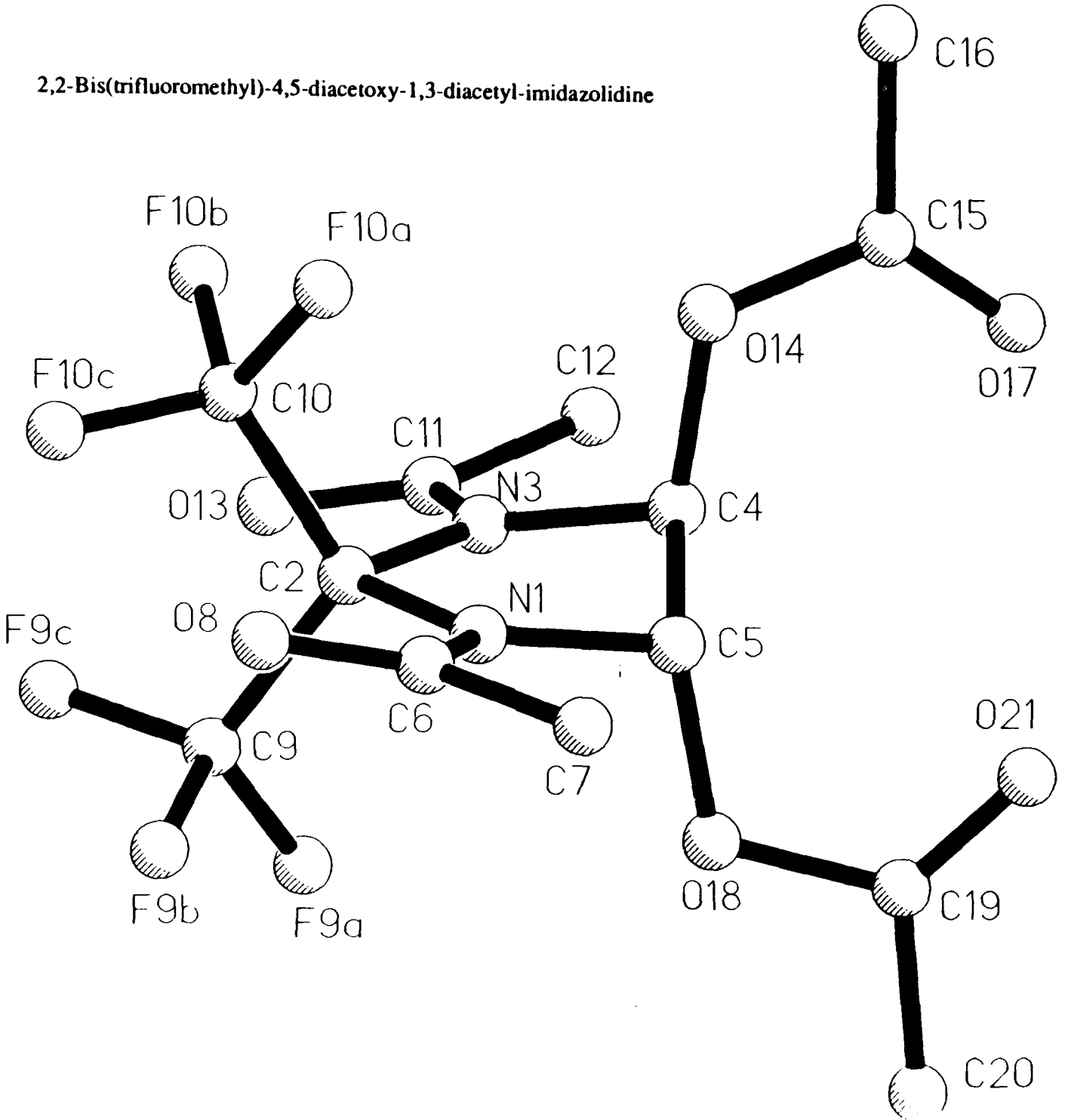


TABLE **1p.** Atom coordinates (x10) and temperature factors (A x10)

atom	x	y	z	U
N(1)	3400(4)	967(2)	2130(2)	47(1)*
C(2)	4293(5)	498(2)	1934(3)	48(2)*
N(3)	3560(4)	-53(2)	2038(2)	50(1)*
C(4)	2498(4)	67(2)	2459(3)	49(2)*
C(5)	2125(4)	717(2)	2219(3)	49(2)*
C(6)	3758(5)	1582(2)	2273(3)	56(2)*
C(7)	2684(6)	1984(2)	2457(4)	76(2)*
O(8)	4876(4)	1751(2)	2250(3)	77(1)*
C(9)	4422(6)	568(3)	991(3)	60(2)*
F(9a)	3336(3)	330(2)	436(2)	83(1)*
F(9b)	4469(4)	1148(1)	783(2)	91(1)*
F(9c)	5551(3)	319(2)	873(2)	84(1)*
C(10)	5751(5)	509(3)	2614(4)	65(2)*
F(10a)	5666(3)	738(2)	3362(2)	81(1)*
F(10b)	6252(3)	-50(2)	2789(2)	94(1)*
F(10c)	6694(3)	816(2)	2369(2)	88(1)*
C(11)	3855(6)	-641(2)	1797(4)	65(2)*
C(12)	3059(6)	-1145(2)	2050(4)	80(2)*
O(13)	4731(4)	-714(2)	1423(3)	89(2)*
O(14)	3094(3)	20(2)	3379(2)	55(1)*
C(15)	2280(5)	-213(2)	3849(3)	57(2)*
C(16)	3086(5)	-242(3)	4776(3)	72(2)*
O(17)	1121(3)	-368(2)	3556(2)	70(1)*
O(18)	1126(3)	749(2)	1385(2)	59(1)*
C(19)	-204(5)	694(2)	1409(4)	64(2)*
C(20)	-1125(6)	731(3)	510(4)	90(2)*
O(21)	-514(4)	622(2)	2061(3)	88(2)*
N(51)	7358(4)	2752(2)	1356(2)	50(1)*
C(52)	7408(4)	2963(2)	507(3)	47(2)*
N(53)	6191(4)	3351(2)	256(2)	46(1)*
C(54)	5277(5)	3243(2)	796(3)	49(2)*
C(55)	6233(5)	3016(2)	1627(3)	49(2)*
C(56)	8220(6)	2296(3)	1810(3)	64(2)*
C(57)	8170(6)	2197(3)	2721(3)	81(2)*
O(58)	9005(4)	2034(2)	1493(3)	82(2)*
C(59)	8774(5)	3345(3)	603(4)	62(2)*
F(59a)	8577(3)	3906(1)	837(2)	82(1)*
F(59b)	9807(3)	3117(2)	1210(2)	84(1)*
F(59c)	9187(3)	3369(2)	-111(2)	82(1)*
C(60)	7265(5)	2427(2)	-151(3)	54(2)*
F(60a)	6625(3)	1961(1)	91(2)	72(1)*
F(60b)	6514(3)	2585(1)	-936(2)	74(1)*
F(60c)	8455(3)	2228(1)	-248(2)	81(1)*
C(61)	5911(5)	3751(2)	-441(3)	54(2)*
C(62)	4613(5)	4106(2)	-607(3)	67(2)*
O(63)	6726(4)	3809(2)	-863(2)	69(1)*
O(64)	4308(3)	2779(1)	400(2)	57(1)*
C(65)	3022(6)	2838(3)	446(4)	72(2)*
C(66)	2117(6)	2340(3)	5(4)	94(2)*
O(67)	2673(4)	3253(2)	813(4)	141(2)*
O(68)	6759(3)	3526(1)	2182(2)	58(1)*
C(69)	6136(5)	3644(3)	2817(3)	61(2)*
C(70)	6783(6)	4170(3)	3341(4)	84(2)*
O(71)	5202(4)	3356(2)	2907(3)	96(2)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalised U tensor

TABLE 2p. Bond lengths (A)

N(1)-C(2)	1.461(6)	N(1)-C(5)	1.440(6)
N(1)-C(6)	1.406(6)	C(2)-N(3)	1.455(6)
C(2)-C(9)	1.563(7)	C(2)-C(10)	1.575(6)
N(3)-C(4)	1.436(7)	N(3)-C(11)	1.410(7)
C(4)-C(5)	1.507(7)	C(4)-O(14)	1.441(5)
C(5)-O(18)	1.444(5)	C(6)-C(7)	1.488(8)
C(6)-O(8)	1.197(6)	C(9)-F(9a)	1.319(6)
C(9)-F(9b)	1.327(7)	C(9)-F(9c)	1.322(7)
C(10)-F(10a)	1.329(7)	C(10)-F(10b)	1.334(7)
C(10)-F(10c)	1.312(7)	C(11)-C(12)	1.490(8)
C(11)-O(13)	1.204(8)	O(14)-C(15)	1.357(7)
C(15)-C(16)	1.492(6)	C(15)-O(17)	1.185(6)
O(18)-C(19)	1.355(7)	C(19)-C(20)	1.493(8)
C(19)-O(21)	1.183(8)	N(51)-C(52)	1.455(6)
N(51)-C(55)	1.442(6)	N(51)-C(56)	1.399(6)
C(52)-N(53)	1.459(6)	C(52)-C(59)	1.586(7)
C(52)-C(60)	1.568(7)	N(53)-C(54)	1.445(7)
N(53)-C(61)	1.394(6)	C(54)-C(55)	1.506(6)
C(54)-O(64)	1.439(5)	C(55)-O(68)	1.444(5)
C(56)-C(57)	1.494(8)	C(56)-O(58)	1.198(8)
C(59)-F(59a)	1.324(6)	C(59)-F(59b)	1.318(6)
C(59)-F(59c)	1.324(7)	C(60)-F(60a)	1.327(6)
C(60)-F(60b)	1.330(5)	C(60)-F(60c)	1.324(6)
C(61)-C(62)	1.486(7)	C(61)-O(63)	1.205(7)
O(64)-C(65)	1.323(7)	C(65)-C(66)	1.480(8)
C(65)-O(67)	1.194(9)	O(68)-C(69)	1.359(7)
C(69)-C(70)	1.478(8)	C(69)-O(71)	1.177(7)

TABLE **3p.** Bond angles (deg.)

C(2)-N(1)-C(5)	111.5(4)	C(2)-N(1)-C(6)	125.1(4)
C(5)-N(1)-C(6)	123.3(4)	N(1)-C(2)-N(3)	102.0(4)
N(1)-C(2)-C(9)	110.8(4)	N(3)-C(2)-C(9)	111.8(4)
N(1)-C(2)-C(10)	110.6(4)	N(3)-C(2)-C(10)	109.9(4)
C(9)-C(2)-C(10)	111.4(4)	C(2)-N(3)-C(4)	111.3(4)
C(2)-N(3)-C(11)	125.9(4)	C(4)-N(3)-C(11)	122.7(4)
N(3)-C(4)-C(5)	102.8(4)	N(3)-C(4)-O(14)	108.3(3)
C(5)-C(4)-O(14)	109.7(4)	N(1)-C(5)-C(4)	102.9(4)
N(1)-C(5)-O(18)	107.7(4)	C(4)-C(5)-O(18)	110.2(3)
N(1)-C(6)-C(7)	116.1(4)	N(1)-C(6)-O(8)	119.8(5)
C(7)-C(6)-O(8)	124.0(5)	C(2)-C(9)-F(9a)	110.3(5)
C(2)-C(9)-F(9b)	111.0(4)	F(9a)-C(9)-F(9b)	107.1(4)
C(2)-C(9)-F(9c)	113.4(4)	F(9a)-C(9)-F(9c)	108.9(5)
F(9b)-C(9)-F(9c)	105.9(5)	C(2)-C(10)-F(10a)	111.0(4)
C(2)-C(10)-F(10b)	111.1(4)	F(10a)-C(10)-F(10b)	105.9(4)
C(2)-C(10)-F(10c)	114.5(4)	F(10a)-C(10)-F(10c)	107.7(4)
F(10b)-C(10)-F(10c)	106.2(5)	N(3)-C(11)-C(12)	116.3(5)
N(3)-C(11)-O(13)	119.8(5)	C(12)-C(11)-O(13)	123.8(5)
C(4)-O(14)-C(15)	116.9(3)	O(14)-C(15)-C(16)	109.0(4)
O(14)-C(15)-O(17)	124.4(4)	C(16)-C(15)-O(17)	126.6(5)
C(5)-O(18)-C(19)	114.5(4)	O(18)-C(19)-C(20)	109.1(5)
O(18)-C(19)-O(21)	122.5(4)	C(20)-C(19)-O(21)	128.4(5)
C(52)-N(51)-C(55)	112.5(3)	C(52)-N(51)-C(56)	123.0(4)
C(55)-N(51)-C(56)	124.1(4)	N(51)-C(52)-N(53)	102.0(4)
N(51)-C(52)-C(59)	109.1(3)	N(53)-C(52)-C(59)	110.8(4)
N(51)-C(52)-C(60)	111.9(4)	N(53)-C(52)-C(60)	109.9(3)
C(59)-C(52)-C(60)	112.7(4)	C(52)-N(53)-C(54)	111.1(4)
C(52)-N(53)-C(61)	124.5(4)	C(54)-N(53)-C(61)	124.3(4)
N(53)-C(54)-C(55)	103.2(4)	N(53)-C(54)-O(64)	108.5(4)
C(55)-C(54)-O(64)	110.4(4)	N(51)-C(55)-C(54)	103.5(4)
N(51)-C(55)-O(68)	108.3(3)	C(54)-C(55)-O(68)	109.1(4)
N(51)-C(56)-C(57)	115.8(5)	N(51)-C(56)-O(58)	120.9(5)
C(57)-C(56)-O(58)	123.2(5)	C(52)-C(59)-F(59a)	109.4(4)
C(52)-C(59)-F(59b)	111.0(4)	F(59a)-C(59)-F(59b)	107.3(4)
C(52)-C(59)-F(59c)	113.9(4)	F(59a)-C(59)-F(59c)	108.4(5)
F(59b)-C(59)-F(59c)	106.7(4)	C(52)-C(60)-F(60a)	110.4(4)
C(52)-C(60)-F(60b)	111.5(4)	F(60a)-C(60)-F(60b)	106.5(4)
C(52)-C(60)-F(60c)	114.1(4)	F(60a)-C(60)-F(60c)	107.8(4)
F(60b)-C(60)-F(60c)	106.0(4)	N(53)-C(61)-C(62)	117.2(5)
N(53)-C(61)-O(63)	120.0(4)	C(62)-C(61)-O(63)	122.8(4)
C(54)-O(64)-C(65)	117.6(4)	O(64)-C(65)-C(66)	112.7(5)
O(64)-C(65)-O(67)	121.7(5)	C(66)-C(65)-O(67)	125.6(6)
C(55)-O(68)-C(69)	116.5(4)	O(68)-C(69)-C(70)	110.9(5)
O(68)-C(69)-O(71)	122.4(5)	C(70)-C(69)-O(71)	126.7(6)

Abstract

2-Acetoxy-3(N-acetamidomethyl-N-nitroso)amino-1,4,-dinitropiperazine, $C_9H_{15}N_7O_8$, M_r = 349.26, orthorhombic, C2/c, a = 19.736(2), b = 14.750(2), c = 10.987(1) Å, V = 3198.3(5) Å^3, Z = 8, D_x = 1.450 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 10.69 cm^{-1}, F(000) = 1456, T = 295 K, Final R = 0.043, wR = 0.054 for 2241 independent reflections.

Experimental

A clear colorless 0.05 x 0.35 x 0.50 mm data crystal was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within 37 ≤ 2θ ≤ 71° used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.57 Å^{-1}, range of hkl : -22 ≤ h ≤ 3>, 0 ≤ k ≤ 16, 12 ≤ l ≤ 12. Standards 14 0 0, 080, 606, monitored every 60 reflections with linear variation of 4.1 % over data collection, θ/2θ mode, scan width [2θ(Kα$_1$) - 1.0] to [2θ(Kα$_2$) + 1.0]°, scan rate a function of count rate (8.0°/min. minimum, 30.0°/min. maximum, 3439 reflections measured, 2550 unique, R_{int} = 0.01, 2241 observed with Fo>3σ(F_o). Data corrected for Lorentz and polarization, but not absorbtion effects . Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. Secondary extinction parameter p = 0.0014(2) in $F_c^* = F_c/[\ 1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. 237 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms,H atoms included using riding model , C-H = 0.96 Å, C-C-H = 109.5° , U(H) = 1.2 U_{eq}(C) . (Δ/σ) max = 0.012, R = 0.043, wR = 0.054, S = 2.075. Final difference Fourier excursions 0.22 and -.018 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1q).

2-Acetoxy-3(N-acetamidomethyl-N-nitroso)amino-1,4,-dinitropiperazine

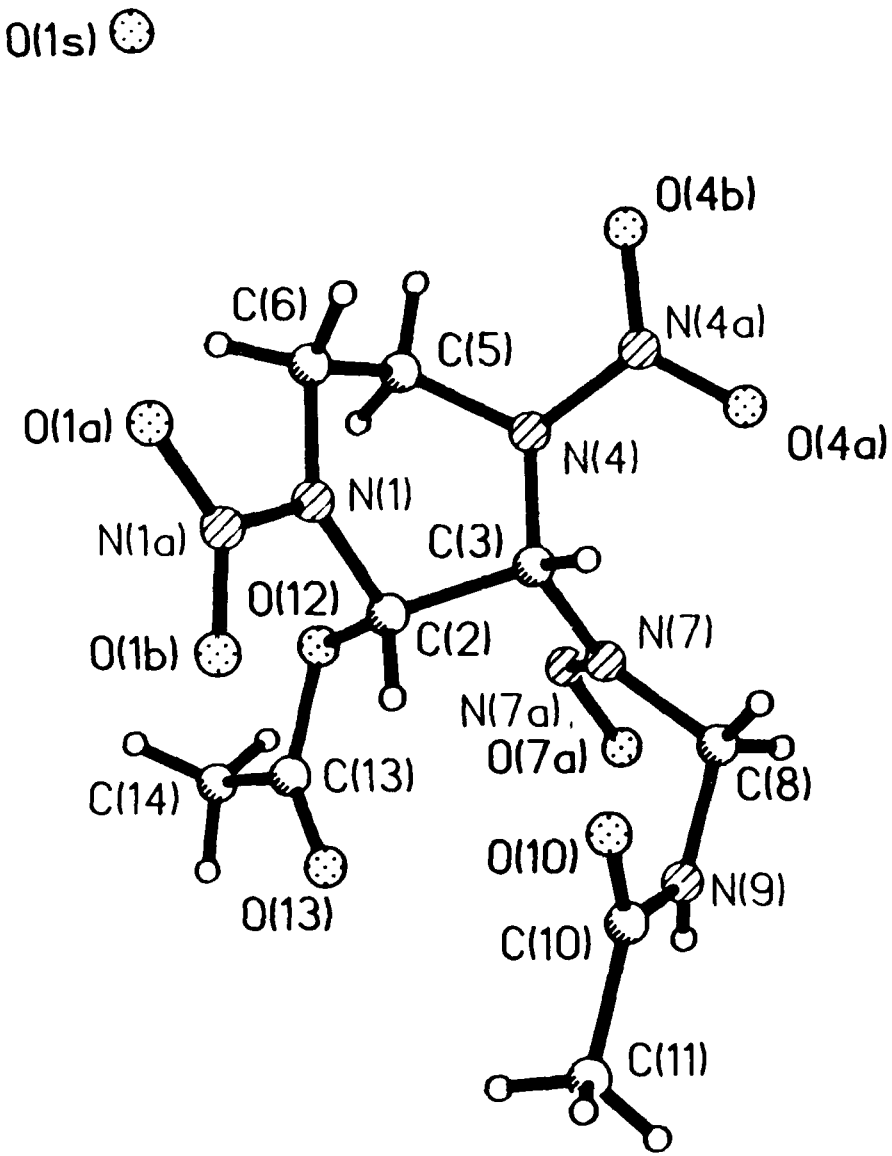


Table 1q. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 x10^3$)

	x	y	z	U(eq)
N(1)	2229(1)	1024(1)	3048(2)	50(1)
N(1A)	2094(1)	384(1)	3902(2)	60(1)
O(1A)	1538(1)	20(1)	3834(2)	84(1)
O(1B)	2519(1)	230(1)	4678(1)	81(1)
C(2)	2884(1)	1441(1)	3022(2)	44(1)
C(3)	2792(1)	2465(1)	2815(2)	39(1)
N(4)	2315(1)	2627(1)	1831(1)	44(1)
N(4A)	1919(1)	3390(1)	1977(2)	54(1)
O(4A)	2147(1)	3983(1)	2634(2)	63(1)
O(4B)	1384(1)	3427(1)	1419(2)	83(1)
C(5)	2028(1)	1848(2)	1194(2)	53(1)
C(6)	1737(1)	1166(2)	2077(2)	60(1)
N(7)	3437(1)	2927(1)	2615(1)	40(1)
N(7A)	3734(1)	2793(1)	1549(1)	51(1)
O(7A)	4288(1)	3179(1)	1452(2)	67(1)
C(8)	3730(1)	3535(1)	3523(2)	47(1)
N(9)	4247(1)	3126(1)	4247(2)	50(1)
C(10)	4095(1)	2598(2)	5210(2)	51(1)
O(10)	3506(1)	2477(1)	5511(1)	68(1)
C(11)	4686(1)	2200(2)	5866(2)	77(1)
O(12)	3263(1)	1032(1)	2061(1)	49(1)
C(13)	3898(1)	727(2)	2322(2)	57(1)
O(13)	4149(1)	840(2)	3295(2)	91(1)
C(14)	4192(1)	272(2)	1248(2)	74(1)
O(1S)	114(8)	305(10)	1180(18)	475(13)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

TABLE 2q. Bond lengths (A)

N(1)-N(1a)	1.357(2)	N(1)-C(2)	1.431(3)
N(1)-C(6)	1.458(3)	N(1a)-O(1a)	1.225(3)
N(1a)-O(1b)	1.218(3)	C(2)-C(3)	1.538(3)
C(2)-O(12)	1.429(2)	C(3)-N(4)	1.453(2)
C(3)-N(7)	1.460(2)	N(4)-N(4a)	1.380(2)
N(4)-C(5)	1.460(3)	N(4a)-O(4a)	1.220(2)
N(4a)-O(4b)	1.222(2)	C(5)-C(6)	1.511(3)
N(7)-N(7a)	1.326(2)	N(7)-C(8)	1.460(2)
N(7a)-O(7a)	1.237(2)	C(8)-N(9)	1.427(3)
N(9)-C(10)	1.348(3)	C(10)-O(10)	1.222(3)
C(10)-C(11)	1.491(3)	O(12)-C(13)	1.361(3)
C(13)-O(13)	1.190(3)	C(13)-C(14)	1.477(4)

TABLE 3q. Bond angles (deg.)

N(1a)-N(1)-C(2)	119.3(2)	N(1a)-N(1)-C(6)	118.3(2)
C(2)-N(1)-C(6)	121.7(2)	N(1)-N(1a)-O(1a)	116.0(2)
N(1)-N(1a)-O(1b)	118.6(2)	O(1a)-N(1a)-O(1b)	125.4(2)
N(1)-C(2)-C(3)	108.6(2)	N(1)-C(2)-O(12)	107.9(1)
C(3)-C(2)-O(12)	111.5(1)	C(2)-C(3)-N(4)	110.4(1)
C(2)-C(3)-N(7)	112.3(1)	N(4)-C(3)-N(7)	112.1(1)
C(3)-N(4)-N(4a)	114.5(1)	C(3)-N(4)-C(5)	118.6(2)
N(4a)-N(4)-C(5)	118.5(2)	N(4)-N(4a)-O(4a)	116.4(2)
N(4)-N(4a)-O(4b)	117.9(2)	O(4a)-N(4a)-O(4b)	125.7(2)
N(4)-C(5)-C(6)	111.4(2)	N(1)-C(6)-C(5)	108.2(2)
C(3)-N(7)-N(7a)	116.7(1)	C(3)-N(7)-C(8)	122.0(1)
N(7a)-N(7)-C(8)	121.3(1)	N(7)-N(7a)-O(7a)	113.5(2)
N(7)-C(8)-N(9)	113.9(2)	C(8)-N(9)-C(10)	121.5(2)
N(9)-C(10)-O(10)	120.6(2)	N(9)-C(10)-C(11)	115.6(2)
O(10)-C(10)-C(11)	123.8(2)	C(2)-O(12)-C(13)	117.8(2)
O(12)-C(13)-O(13)	121.8(2)	O(12)-C(13)-C(14)	110.1(2)
O(13)-C(13)-C(14)	128.1(2)		

Abstract

1,1,3,3-Tetranitrocyclobutane, $C_4H_4N_4O_8$, M_r = 236.10, $P\bar{1}$, triclinic a = 6.301(1), b = 7.857(1), c = 8.736(1) Å, α = 85.88(1), β = 84.62(1), γ = 85.13(1)°, V = 428.2(1) Å^3, Z = 2, D_x = 1.83 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 15.60 cm^{-1}, F(000) = 240, T = 295 K, Final R = 0.037, wR = 0.045 for 1146 independent reflections. There are two half molecules in the asymmetric unit, each with an inversion center at the center of the butane ring.

Experimental

A clear colorless 0.05 x 0.10 x 0.35 mm data crystal recrystallized from methylene chloride/chloroform solvent mixture was provided by K. Baum of Fluorochem Inc., Azusa CA.. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within 61 ≤ 2θ ≤ 88° used for determining lattice parameters. $(\sin(\theta)/\lambda)_{max}$ = 0.56 Å^{-1}, range of hkl : -7 ≤ h ≤ 3, -8 ≤ k ≤ 8, -9 ≤ ≤ 9. Standards 212, 113, 042, monitored every 60 reflections with random variation of 2.0 % over data collection, θ/2θ mode, scan width [2θ(Kα_1) - 1.0] to [2θ(Kα_2) + 1.0]°, scan rate a function of count rate (4.0°/min. minimum, 30.0°/min. maximum, 2587 reflections measured, 1271 unique, R_{int} = 0.009, 1146 observed with Fo>3σ(F_o). Data corrected for Lorentz and polarization, but not absorbtion effects. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. Secondary extinction parameter p = 0.063(5) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. 162 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, isotropic thermal parameters for H atoms . (Δ/σ) max = 0.004, R = 0.037, wR = 0.045, S = 1.90 Final difference Fourier excursions 0.20 and -0.18 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1r).

1, 1, 3, 3-TETRANITROCYCLOBUTANE

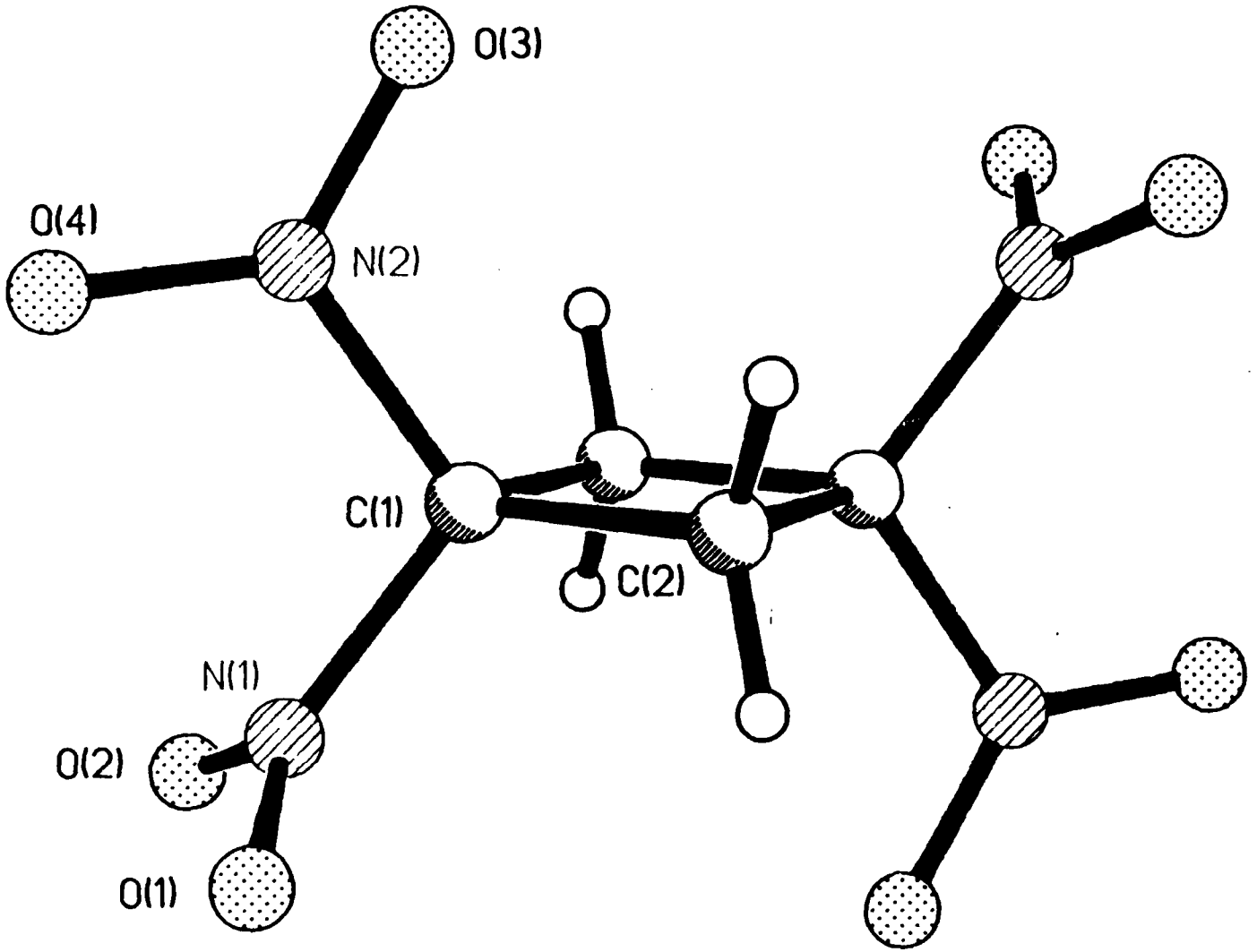


Table 1r. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($A^2 x10^3$)

	x	y	z	U(eq)
C(1)	-528(3)	-148(2)	1198(2)	30(1)
N(1)	678(3)	-991(2)	2483(2)	41(1)
O(1)	1756(3)	-119(2)	3125(2)	70(1)
O(2)	495(3)	-2507(2)	2779(2)	62(1)
N(2)	-2699(3)	476(2)	1912(2)	42(1)
O(3)	-3911(3)	1122(2)	999(2)	69(1)
O(4)	-3069(3)	317(2)	3294(2)	70(1)
C(2)	614(3)	1231(2)	185(2)	34(1)
C(1')	-5236(3)	-5061(2)	3809(2)	30(1)
N(1')	-3851(3)	-4436(2)	2419(2)	37(1)
O(1')	-2438(3)	-5439(2)	1904(2)	56(1)
O(2')	-4263(3)	-2977(2)	1924(2)	56(1)
N(2')	-7086(3)	-5810(2)	3187(2)	40(1)
O(3')	-8374(3)	-6374(2)	4173(2)	69(1)
O(4')	-7164(3)	-5805(2)	1822(2)	61(1)
C(2')	-4080(4)	-6250(3)	4991(2)	36(1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

TABLE 2r. Bond lengths (A)

C(1)-N(1)	1.498(2)	C(1)-N(2)	1.507(2)
C(1)-C(2)	1.533(3)	C(1)-C(2a)	1.534(3)
N(1)-O(1)	1.204(3)	N(1)-O(2)	1.214(2)
N(2)-O(3)	1.211(3)	N(2)-O(4)	1.207(3)
C(2)-C(1a)	1.534(3)	C(1')-N(1')	1.505(2)
C(1')-N(2')	1.510(3)	C(1')-C(2')	1.535(3)
C(1')-C(2'a)	1.530(3)	N(1')-O(1')	1.213(2)
N(1')-O(2')	1.212(2)	N(2')-O(3')	1.214(2)
N(2')-O(4')	1.198(3)	C(2')-C(1'a)	1.530(3)

TABLE 3r. Bond angles (deg.)

N(1)-C(1)-N(2)	106.6(1)	N(1)-C(1)-C(2)	115.6(2)
N(2)-C(1)-C(2)	113.7(1)	N(1)-C(1)-C(2a)	115.5(1)
N(2)-C(1)-C(2a)	113.7(2)	C(2)-C(1)-C(2a)	91.6(1)
C(1)-N(1)-O(1)	117.7(2)	C(1)-N(1)-O(2)	116.8(2)
O(1)-N(1)-O(2)	125.5(2)	C(1)-N(2)-O(3)	114.5(2)
C(1)-N(2)-O(4)	119.1(2)	O(3)-N(2)-O(4)	126.4(2)
C(1)-C(2)-C(1a)	88.4(1)	N(1')-C(1')-N(2')	105.8(1)
N(1')-C(1')-C(2')	115.5(2)	N(2')-C(1')-C(2')	114.5(2)
N(1')-C(1')-C(2'a)	115.8(2)	N(2')-C(1')-C(2'a)	113.8(2)
C(2')-C(1')-C(2'a)	91.4(1)	C(1')-N(1')-O(1')	117.0(2)
C(1')-N(1')-O(2')	116.6(2)	O(1')-N(1')-O(2')	126.4(2)
C(1')-N(2')-O(3')	114.2(2)	C(1')-N(2')-O(4')	119.4(2)
O(3')-N(2')-O(4')	126.4(2)	C(1')-C(2')-C(1'a)	88.6(1)

Abstract

Bimethylene trisacetamide, $C_8H_{15}N_3O_3$, M_r = 201.23, orthorhombic, $P2_12_12_1$, a = 7.311(1), b = 9,581(2), c = 15.105(2) Å, V = 1058.1(2) Å^3, Z = 4, D_x = 1.263 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 7.80 cm^{-1}, F(000) = 432, T = 295 K, Final R = 0.040, wR = 0.047 for 948 independent reflections.

Experimental

A clear colorless 0.15 x 0.15 x 0.20 mm data crystal was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within $40 \leq 2\theta \leq 60°$ used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.59 Å^{-1}, range of hkl : $0 \leq h \leq 8$, $0 \leq k \leq 10$, $-16 \leq l \leq 0$. Standards 040, 004, 312, monitored every 60 reflections with linear variation of 2.2 % over data collection, $\theta/2\theta$ mode, scan width [$2\theta(K\alpha_1)$ - 1.0] to [$2\theta(K\alpha_2)$ + 1.0]°, scan rate a function of count rate (4°/min. minimum, 30°/min. maximum, 1089 reflections measured, 1070 unique, no equilalents measured, 948 observed with $Fo > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorbtion effects. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.00023. 160 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, methyl hydrogens included using riding model , C-H = 0.96 Å, C-C-H = 109.5° , $U(H) = 1.2U_{eq}(C)$, other H atoms refined with isotropuc thermal parameters. (Δ/σ) max = 0.24, R = 0.040, wR = 0.047, S = 1.83. Final difference Fourier excursions 0.16 and -0.14 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1s).

Bimethylene trisacetamide

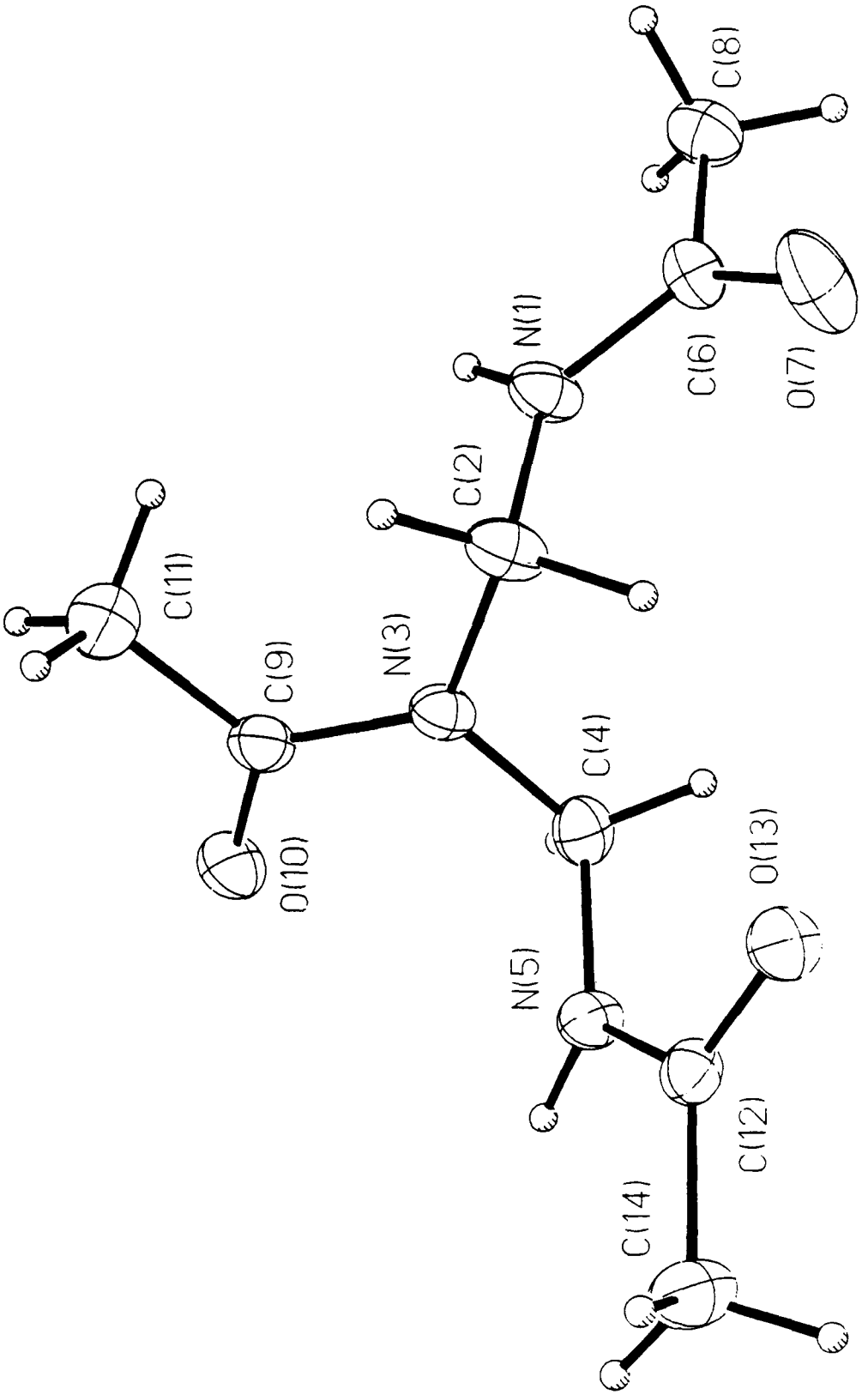


Table 1s. Atom coordinates ($x10^4$) and thermal parameters ($Å^2 \times 10^3$)

atom	x	y	z	U
N(1)	4862(4)	417(3)	4469(2)	56(1)*
C(2)	6811(5)	411(3)	4319(3)	58(1)*
N(3)	7443(3)	-996(2)	4121(2)	47(1)*
C(4)	7334(5)	-1488(3)	3203(2)	52(1)*
N(5)	9098(4)	-1637(2)	2797(2)	48(1)*
C(6)	3753(4)	1418(3)	4172(2)	52(1)*
O(7)	4331(4)	2428(2)	3768(2)	91(1)*
C(8)	1777(5)	1253(4)	4396(3)	67(1)*
C(9)	7995(4)	-1919(3)	4748(2)	52(1)*
O(10)	8398(4)	-3104(2)	4526(2)	69(1)*
C(11)	8115(6)	-1461(4)	5695(2)	78(1)*
C(12)	9996(5)	-540(3)	2462(2)	47(1)*
O(13)	9337(3)	649(2)	2499(1)	58(1)*
C(14)	11827(6)	-834(4)	2061(3)	72(1)*

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

TABLE 2s. Bond lengths (A)

N(1)-C(2)	1.443(5)	N(1)-C(6)	1.334(4)
C(2)-N(3)	1.456(4)	N(3)-C(4)	1.466(4)
N(3)-C(9)	1.357(4)	C(4)-N(5)	1.435(4)
N(5)-C(12)	1.339(4)	C(6)-O(7)	1.220(4)
C(6)-C(8)	1.492(5)	C(9)-O(10)	1.220(4)
C(9)-C(11)	1.500(5)	C(12)-O(13)	1.238(3)
C(12)-C(14)	1.496(5)		

TABLE 3s. Bond angles (deg.)

C(2)-N(1)-C(6)	123.4(3)	N(1)-C(2)-N(3)	110.4(2)
C(2)-N(3)-C(4)	118.3(2)	C(2)-N(3)-C(9)	123.7(3)
C(4)-N(3)-C(9)	117.8(2)	N(3)-C(4)-N(5)	112.7(3)
C(4)-N(5)-C(12)	121.6(3)	N(1)-C(6)-O(7)	121.9(3)
N(1)-C(6)-C(8)	115.9(3)	O(7)-C(6)-C(8)	122.2(3)
N(3)-C(9)-O(10)	119.1(3)	N(3)-C(9)-C(11)	119.5(3)
O(10)-C(9)-C(11)	121.4(3)	N(5)-C(12)-O(13)	120.9(3)
N(5)-C(12)-C(14)	116.4(3)	O(13)-C(12)-C(14)	122.7(3)

Abstract

5H,10H-di-tetrazolo[1,5-a;1,5-d]piperazine, $C_4H_4N_8$, M_r = 164.13, monoclinic, $P2_1/n$, a = 6.439(1), b = 6.400(1), c = 7.807(1) Å, β =98.95(1)°, V = 317.8(1) Å^3, Z = 4, D_x = 1.715 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 10.5 cm^{-1}, F(000) = 168, T = 295 K, Final R = 0.039, wR = 0.059 for 449 independent reflections.

Experimental

A clear colorless 0.10 x 0.15 x 0.40 mm data crystal was provided by R. Willer of Morton Thiokol. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within $45 \leq 2\theta \leq 80°$ used for determining lattice parameters . $(\sin(\theta)/\lambda)_{max}$ = 0.55 Å^{-1}, range of hkl : $0 \leq h \leq 7$, $0 \leq k \leq 7$, $-8 \leq \leq 8$. Standards 400, 040, 004, monitored every 60 reflections with random variation of 1.3 % over data collection, $\theta/2\theta$ mode, scan width [$2\theta(K\alpha_1)$ - 1.0] to [$2\theta(K\alpha_2)$ + 1.0]°, scan rate a function of count rate (4°/min. minimum, 30°/min. maximum, 603 reflections measured, 470 unique, R_{int} = 0.01, 449 observed with $Fo>3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorbtion effects. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 0.0004. 64 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, isotropic thermal parameters for H atoms . (Δ/σ) max = 0.003, R = 0.039, wR = 0.059, S = 2.288. Final difference Fourier excursions 0.23 and -0.19 eÅ^{-3}. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, 2, and 3, atom coordinates, bond distances and angles, follows that shown in Fig.(1t).

Table 1t. Atom coordinates ($x10^4$) and thermal parameters ($Å^2 \times 10^3$)

atom	x	y	z	U
N(1)	9877(3)	3166(3)	4141(2)	34(1)*
N(2)	9398(3)	1368(3)	3293(2)	40(1)*
N(3)	7539(3)	838(4)	3651(3)	49(1)*
N(4)	6816(3)	2253(3)	4681(2)	43(1)*
C(5)	8311(3)	3703(3)	4976(2)	27(1)*
C(6)	8218(3)	5640(4)	5980(3)	38(1)*

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

TABLE 2t. Bond lengths (A)

N(1)-N(2)	1.340(2)	N(1)-C(5)	1.328(3)
N(1)-C(6a)	1.460(3)	N(2)-N(3)	1.315(3)
N(3)-N(4)	1.341(3)	N(4)-C(5)	1.331(3)
C(5)-C(6)	1.473(3)	C(6)-N(1a)	1.460(3)

TABLE 3t. Bond angles (deg.)

N(2)-N(1)-C(5)	109.3(2)	N(2)-N(1)-C(6a)	123.3(2)
C(5)-N(1)-C(6a)	127.3(2)	N(1)-N(2)-N(3)	105.3(2)
N(2)-N(3)-N(4)	111.4(2)	N(3)-N(4)-C(5)	105.4(2)
N(1)-C(5)-N(4)	108.6(2)	N(1)-C(5)-C(6)	125.1(2)
N(4)-C(5)-C(6)	126.2(2)	C(5)-C(6)-N(1a)	107.5(2)

Abstract

1,4-Dinitro-2-acetoxy-3-hydroxy-1,4-diazacyclohexane, $C_6H_{10}N_4O_7$, M_r = 250.17, monoclinic, $P2_1/c$, a = 13.588(2), b = 7.276(1), c = 10.830(1) Å, β = 109.75(1)°, V = 1007.7(2) Å^3, Z = 4, D_x = 1.65 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 12.81 cm^{-1}, F(000) = 520, T = 295 K, Final R = 0.029, wR = 0.028 for 1239 independent reflections.

Experimental

A clear colorless 0.10 x 0.15 x 0.40 mm data crystal was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within $15 \leq 2\theta \leq 81°$ used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.55 Å^{-1}, range of hkl : $-14 \leq h \leq 14$, $0 \leq k \leq 7$, $0 \leq l \leq 10$. Standards 13 0 0, 240, 008, monitored every 60 reflections with random variation of 2.0 % over data collection, $\theta/2\theta$ mode, scan width [$2\theta(K\alpha_1)$ - 1.0] to [$2\theta(K\alpha_2)$ + 1.0]°, scan rate a function of count rate (10.0°/min. minimum, 30.0°/min. maximum, 1686 reflections measured, 1551 unique, R_{int} = 0.008, 1239 observed with $Fo > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorbtion effects . Structure solved by direct methods. The least-squares refinement used program MULTAN. $\Sigma w(|F_o|-|F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, g = 1.00. Secondary extinction parameter p = 0.00002(1) in $F_c^* = F_c/[1.0 + (p)I_c]$. 185 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included with $U(H) = 1.1\ U_{eq}(C)$. (Δ/σ) max = 0.004, R = 0.029, wR = 0.028, S = 0.60. Final difference Fourier excursions 0.13 and -0.18 eÅ^3. Atomic scattering factors from International Tables for X-ray Crystallography (1974).* Atom numbering for tables 1, and 2, atom coordinates, bond distances and angles, follows that shown in Fig.(1u).

1,4-Dinitro-2-acetoxy-3-hydroxy-1,4-diazacyclohexane

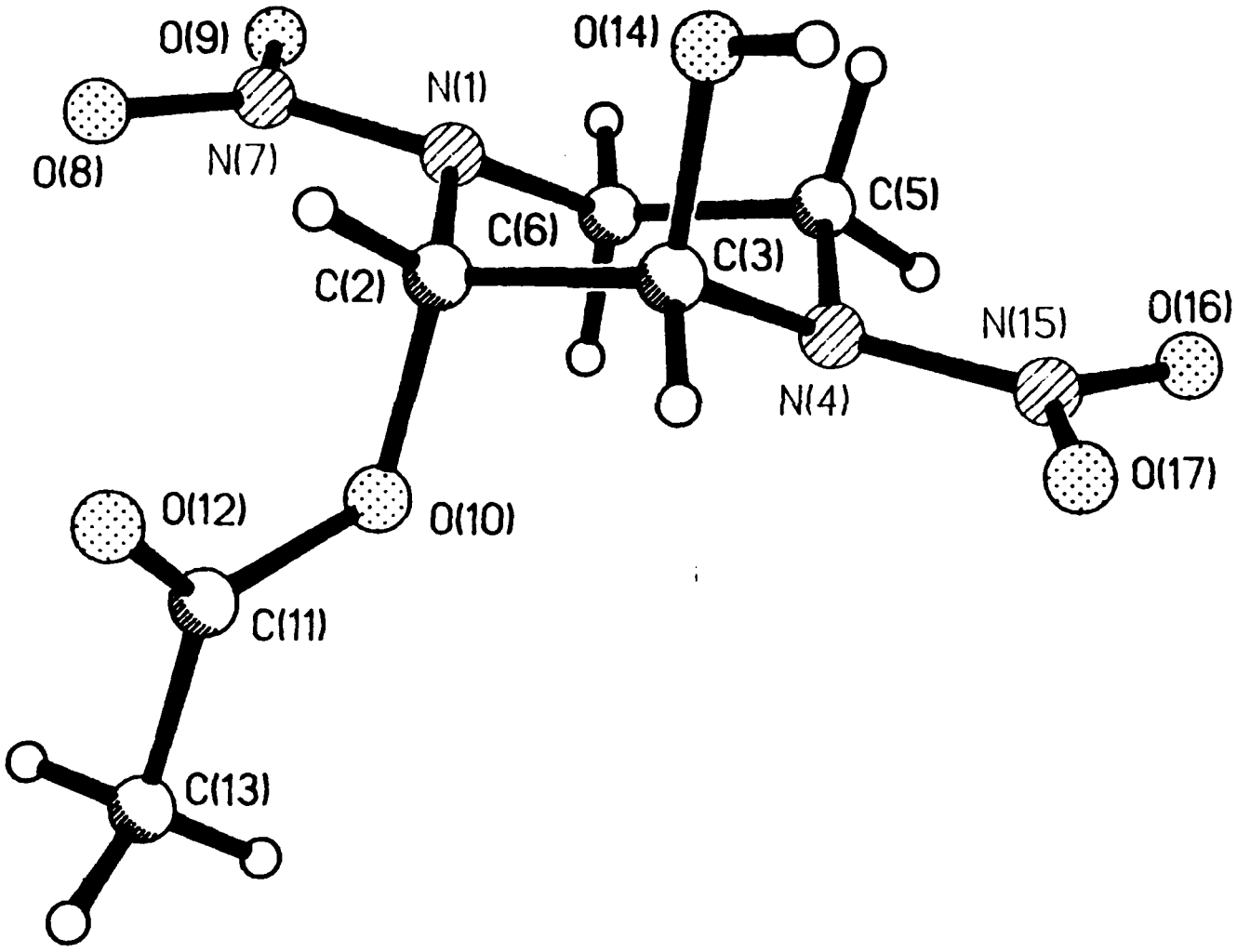


Table 1α.

Positional Parameters and Their Estimated Standard Deviations

Atom	x	y	z	B(A2)
N1	0.1914(1)	0.5908(2)	0.1245(2)	2.75(4)
C2	0.2921(2)	0.5032(3)	0.1756(2)	2.58(4)
C3	0.2780(2)	0.2971(3)	0.1918(2)	2.66(5)
N4	0.2028(1)	0.2696(2)	0.2602(2)	2.68(4)
C5	0.1007(2)	0.3583(3)	0.2048(3)	4.00(6)
C6	0.1170(2)	0.5613(3)	0.1934(2)	3.83(5)
N7	0.1915(1)	0.7648(3)	0.0732(2)	3.39(4)
O8	0.2713(1)	0.8161(2)	0.0545(2)	4.28(4)
O9	0.1110(1)	0.8544(2)	0.0469(2)	4.94(5)
O10	0.3471(1)	0.5819(2)	0.3019(1)	2.86(3)
C11	0.4481(2)	0.6338(3)	0.3239(2)	3.04(5)
O12	0.4937(1)	0.5970(3)	0.2507(2)	5.08(4)
C13	0.4899(2)	0.7396(4)	0.4488(2)	3.84(6)
O14	0.2431(1)	0.2238(2)	0.0645(1)	3.77(4)
N15	0.2000(1)	0.0920(3)	0.3056(2)	3.15(4)
O16	0.1201(1)	0.0413(2)	0.3227(2)	4.31(4)
O17	0.2796(1)	0.0004(2)	0.3293(2)	4.53(4)

nisotropically refined atoms are given in the form of the
sotropic equivalent displacement parameter defined as:
4/3) * [a2*β(1,1) + b2*β(2,2) + c2*β(3,3) + ab(cos gamma)*β(1,2)
ac(cos beta)*β(1,3) + bc(cos alpha)*β(2,3)]

Table 8u.

Bond Distances in Angstroms

Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance
N1	C2	1.440(2)	C3	O14	1.403(2)	O10	C11	1.364(2)
N1	C6	1.462(3)	N4	C5	1.463(3)	C11	O12	1.191(3)
N1	N7	1.383(3)	N4	N15	1.387(3)	C11	C13	1.492(3)
C2	C3	1.529(3)	C5	C6	1.505(3)	N15	O16	1.216(3)
C2	O10	1.437(2)	N7	O8	1.226(3)	N15	O17	1.221(2)
C3	N4	1.464(3)	N7	O9	1.221(2)			

Bond Angles in Degrees

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
C2	N1	C6	118.1(2)	N4	C3	O14	112.5(2)	O8	N7	O9	124.9(2)
C2	N1	N7	115.5(2)	C3	N4	C5	117.4(2)	C2	O10	C11	116.5(2)
C6	N1	N7	115.7(2)	C3	N4	N15	114.5(2)	O10	C11	O12	122.7(2)
N1	C2	C3	109.8(2)	C5	N4	N15	114.8(2)	O10	C11	C13	110.7(2)
N1	C2	O10	108.1(2)	N4	C5	C6	108.7(2)	O12	C11	C13	126.6(2)
C3	C2	O10	109.1(2)	N1	C6	C5	109.4(2)	N4	N15	O16	117.9(2)
C2	C3	N4	109.1(2)	N1	N7	O8	117.8(2)	N4	N15	O17	117.0(2)
C2	C3	O14	105.9(2)	N1	N7	O9	117.4(2)	O16	N15	O17	125.0(2)

Numbers in parentheses are estimated standard deviations in the least significant digits.

Abstract

1,4-Butanediammonium dinitrate, $C_4H_8(NH_3^+)_2 \cdot (NO_3^-)_2$, F.W. = 212.16. triclinic, $P\bar{1}$, a = 5.505(1), b = 8.551(1), c = 10.567(2) Å, α = 92.40(2), β = 91.02(2), γ = 102.04(2)°, V = 485.9(2) Å^3, Z = 2, D_x = 1.450 Mg m^{-3}, λ(Cu Kα) = 1.54178 Å, μ = 11.4 cm^{-1}, F(000) = 224, T = 295 K, Final R = 0.050, wR = 0.061, for 1342 independent reflections. The asymmetric unit is composed of two half formula units con- sisting of a nitrate anion (NT^-) and half of the butanediammonium cation (BDA^+). Each of the half cations of BDA^+ is located on an inversion center and one of them exhibits a disorder in which the central methylene carbon occupies alternate positions C2' and C2" with occupancies of 75 and 25% respectively.

Experimental

A clear colorless 0.05 x 0.10 x 0.70 mm data crystal recrystallized from aqueous ethanol was provided by R. McKenny of the Air Arament Laboratory. Automated Nicolet R3m diffractometer with incident beam monochromator. 25 centered reflections within $43 \leq 2\theta \leq 70°$ used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ = 0.56 Å^{-1}, range of hkl : $-6 \leq h \leq 0$, $-9 \leq k \leq 9$, $-11 \leq l \leq 11$. Standards 300, 040, 006, monitored every 60 reflections with random variation of 2.5 % over data collection, $\theta/2\theta$ mode, scan width $(2.0 + \Delta_{\alpha 1 \alpha 2})°$, scan rate a function of count rate (10°/min. minimum, 30°/min. maximum). 1693 reflections measured, 1438 unique, R_{int} = 0.010, 1342 observed with $Fo > 3\sigma(Fo)$. Data corrected for Lorentz and polarization, but not absorbtion effects. Structure solved by direct methods. The least-squares refinement used program SHELXTL (Sheldrick 1980). $\Sigma w(|Fo|-|Fc|)^2$ minimized where $w = 1/[\sigma^2(|Fo|) + g.(Fo)^2]$, g = 0.00023. 201 parameters refined : atom coordinates, anisotropic thermal parameters for all non-H atoms, isotropic thermal parameters for all H . (Δ/σ) max = 0.006, R = 0.050, wR = 0.061, S = 2.607. Final difference Fourier excursions 0.34 and -0.33 eÅ^3. Atomic scattering factors from International Tables for X-ray Crystallography (1974).*

Atom numbering for tables 1 and 2, atom coordinates, bond distances and angles, follows that shown in Fig.(1v).

AD-A185 462 X-RAY STRUCTURE ANALYSES AND MOLECULAR MMCHANICS ANALYSES OF DENSE ENERGETIC MATERIALS(U) NAVAL RESEARCH LAB WASHINGTON DC R GILARDI ET AL. 01 JUL 87 2/2

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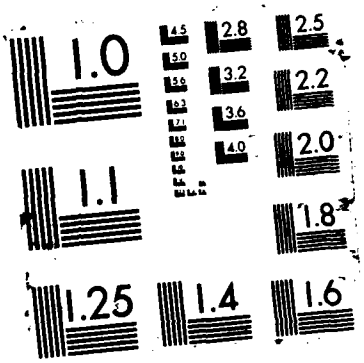
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6.3
7.1
8.0

1,4-Butanediammonium dinitrate

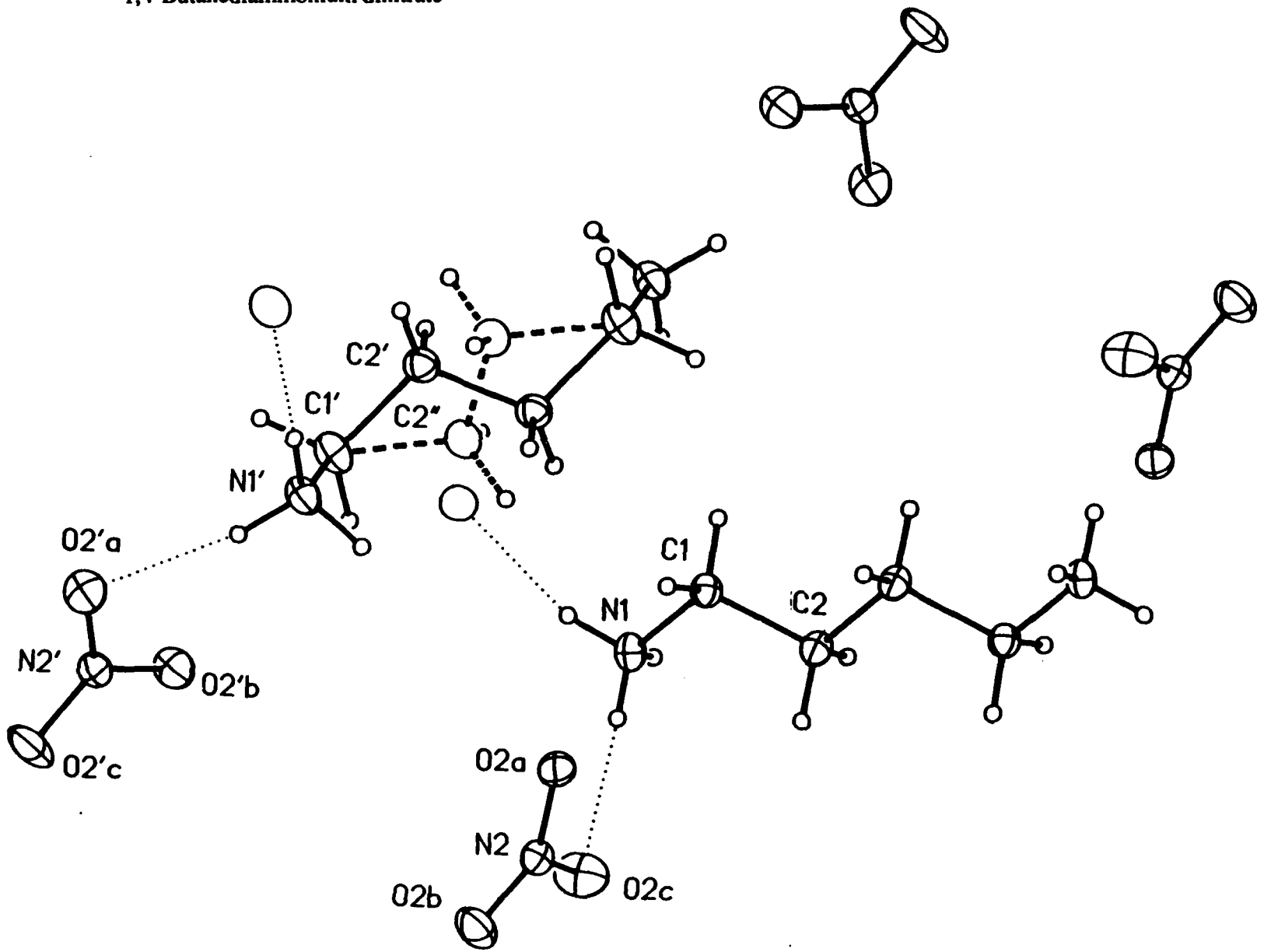

Table 1v. Atom coordinates ($x10^4$) and thermal parameters ($Å^2 \times 10^3$)

atom	x	y	z	U_{eq}*
N(1)	2216(3)	7169(2)	8883(2)	40(1)
C(1)	132(4)	7951(2)	9218(2)	38(1)
C(2)	1062(4)	9592(2)	9864(2)	40(1)
N(1')	880(4)	2502(2)	6303(2)	50(1)
C(1')	1511(5)	3135(3)	5045(2)	55(1)
C(2')	-228(8)	4200(5)	4629(3)	45(1)
C(2")	1322(24)	4973(15)	5058(10)	44(4)
N(2)	3873(3)	5811(2)	11707(2)	40(1)
O(2a)	1615(3)	5794(2)	11574(2)	48(1)
O(2b)	4650(4)	4973(2)	12480(2)	66(1)
O(2c)	5360(3)	6709(3)	11051(2)	75(1)
N(2')	4848(3)	19(2)	6718(2)	41(1)
O(2'a)	2552(3)	-444(2)	6778(2)	60(1)
O(2'b)	5655(3)	1309(2)	6206(2)	58(1)
O(2'c)	6232(4)	-758(3)	7169(2)	78(1)

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2v. Bond Lengths (Å) , bond angles (deg.) and hydrogen bond parameters.

N(1)-C(1)	1.485(3)	C(1)-C(2)	1.511(3)
C(2)-C(2a)	1.515(5)	N(1')-C(1')	1.476(3)
C(1')-C(2')	1.524(5)	C(1')-C(2")	1.596(13)
C(2")-C(2"a)	1.469(27)	C(2')-C(2'a)	1.519(8)
N(2)-O(2a)	1.245(2)	N(2)-O(2b)	1.236(3)
N(2)-O(2c)	1.243(2)	N(2')-O(2'a)	1.247(2)
N(2')-O(2'b)	1.249(2)	N(2')-O(2'c)	1.219(3)
N(1)-C(1)-C(2)	111.5(2)	C(1)-C(2)-C(2a)	111.4(2)
N(1')-C(2')-C(3')	111.6(2)	N(1')-C(2')-C(2")	109.3(4)
C(1')-C(2')-C(2'a)	112.8(4)	C(1')-C(2")-C(2"a)	107.6(11)
O(2a)-N(2)-O(2b)	121.6(2)	O(2a)-N(2)-O(2c)	118.5(2)
O(2b)-N(2)-O(2c)	119.9(2)	O(2a')-N(2')-O(2b')	117.9(2)
O(2a')-N(2')-O(2c')	120.1(2)	O(2b')-N(2')-O(2c')	122.0(2)

N(1)··O(2a)`	2.955(3)	H(1c)··O(2a)`	2.15(1)	∠ N(1)-H(1c)··O(2a)`	151.4(1.1)°
N(1)··O(2c)	2.936(3)	H(1b)··O(2c)	2.08(1)	∠ N(1)-H(1b)··O(2c)	176.9(1.2)°
N(1')··O(2'a)	2.915(3)	H(1'c)··O(2'a)	2.00(1)	∠ N(1')-H(1'c)··O(2'a)	176.5(1.2)°
N(1')··O(2'b)`	2.843(3)	H(1'b)··O(2'b)`	1.89(1)	∠ N(1')-H(1'b)··O(2'b)`	166.7(1.1)°

Published Articles

The Crystal Structure of 4-Benzamido-1-benzoyl-2,3-didehydro-1,2-4-triazolidine.
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Structure of a Substituted Isoxazoline.
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R. Gilardi and C. George; Acta Cryst. **C43**, 363-364 (1987).

Synthesis of 3,5,12-Triazawurtzitanes (3,5,12-Triazatetracyclo[5.3.1.1.^{2,6}.0^{4,9}]dodecanes) .
Nielsen, A. T., Christian, S. L., Moore, D. W., Gilardi, R. and George, C. ;
J. Org. Chem. **52**, 1656-1662 (1987).

Synthesis and Structure of Some Peri-Substituted 2,4,6,8-Tetraazabicyclo[3.3.0]octanes.
W.M. Koppes, M. Chaykovsky, H. G. Adolph, R. Gilardi and C. George;
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Structure of *N*-(4-Amino-3-furazyl)-2,2,2-trichloro-*N'*-methoxyacetamidine.
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Baeyer-Villiger Oxidation of Pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8-11-dione.
Surapaneni , C. R.; and Gilardi R., J. Org. Chem. **51**, 2382-2385(1986).

3,3-Bis(methylnitraminomethyl)oxetane(I) and 3,3-Bis(nitratomethyl)oxetane(II).
George, C. and Gilardi, R.; Acta Cryst. **C42**, 1161-1164 (1986).

Submitted Articles

The Crystal Structure of a Linear Pentaazanonane; C. George and R. Gilardi; (Acta Cryst. C)

The Crystal Structure of an Aliphatic Trinitramine; R. Gilardi and C. George; (Acta Cryst. C)

The Crystal Structure of a Pentaaza Cage Compound; C. George (Acta Cryst. C).

The Crystal Structure of a Substituted Hexahydrotriazine;
C. George and R.Gilardi; (Acta Cryst. C)

Structure of a Pentaazabicyclononane; R. Gilardi; Acta Cryst. (1987), C43, ???-???.

The Crystal Structure of a Dinitrodiamino Compound; R Gilardi and C. George (Acta Cryst. C)

The Crystal Structure of Dinitro-1,4-glycouril; J. Boileau, E. Wilmer, R. Gilardi, M. Stinecipher,
R. Gallo and M. Pierrot (Acta Cryst. C)

Structure of 1,4-Dinitro-2-acetoxy-3-hydroxy-1,4-diazacyclohexane .
J. L. Flippen-Anderson, R. Gilardi and C. George. (Acta Cryst C).

1,6-Dimethyl-1α,4α,4aα,5α,8β,8aα-hexahydro-1,4-methanonaphthalene-5,8-diol, $C_{13}H_{18}O_2$
J. L. Flippen-Anderson, R. Gilardi and C. George. (Acta Cryst C).

syn-8-*syn*-13-*bis*(Benzoyloxy)heptacyclo- [7.6.0.$0^{2,7}$.$0^{4,14}$.$0^{5,12}$.$0^{6,10}$.$0^{11,15}$]pentadecane-3-one, $C_{29}H_{24}O_5$. J. L. Flippen-Anderson, R. Gilardi, C. George, A. P. Marchand and A. D. Earlywine. (Acta Cryst C).

A Novel Rearrangement in the 1,3-*bis*-Homocubyl Ring System. A. P. Marchand, J. L. Flippen-Anderson, R. Gilardi,C. George et al.; J. Chem. Soc. Chem. Comm. (accepted).

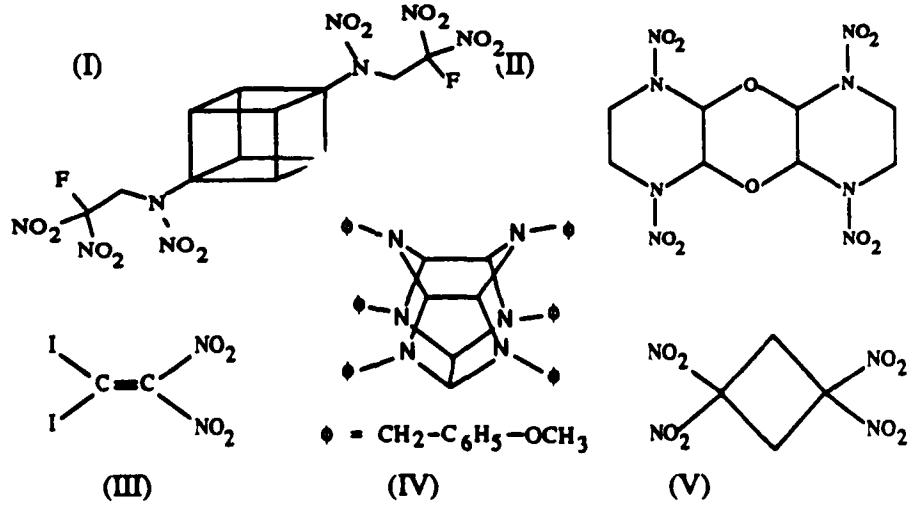
Synthesis of Nitro-substituted 2,3,4,8-Tetraphenylpentacyclo[5.3.0.$0^{2,5}$.$0^{3,9}$.$0^{4,8}$]decanes.
A. P. Marchand, J. L. Flippen-Anderson, R. Gilardi,C. George et al.; J. Org. Chem.

Several Unusual Crowded or Strained Molecules.

Richard Gilardi, Clifford F. George and Judith L. Flippen-Anderson; Laboratory for the Structure of Matter, Naval Research Laboratory, Washington, D.C. 20375-5001

Synthetic chemists interested in energetic materials employ extremely potent reaction conditions and often discover new compounds which are unusually crowded or strained. The structures of these molecules may be of value to molecular and quantum mechanics studies, whether or not the products are useful as propellants or explosives.

(I) is a substituted cubane; the cubane cage is highly strained, and is a good source of energy, but adding substituents at more than one or two positions has proved to be extremely difficult. In this case, energetically substituted side-chains were added to two corners, and the result is fairly stable. The fused-ring nucleus in (II) is a saturated heterocycle in which all three rings are chair-shaped when the nitrogens are alkyl-substituted, but the end rings are shallow boats on the nitro-compound shown here. (III) and (V) represent *gem*-dinitro compounds that are, somewhat surprisingly, quite stable. (III) has a density of *3.07 g/cc* due to the presence of the heavy iodine atoms and the complete absence of hydrogen atoms in the molecule. (IV) is a new hexa-azasubstituted cage compound known as hexa-aza-isowurtzitane; its topology was first established by this X-ray diffraction analysis.

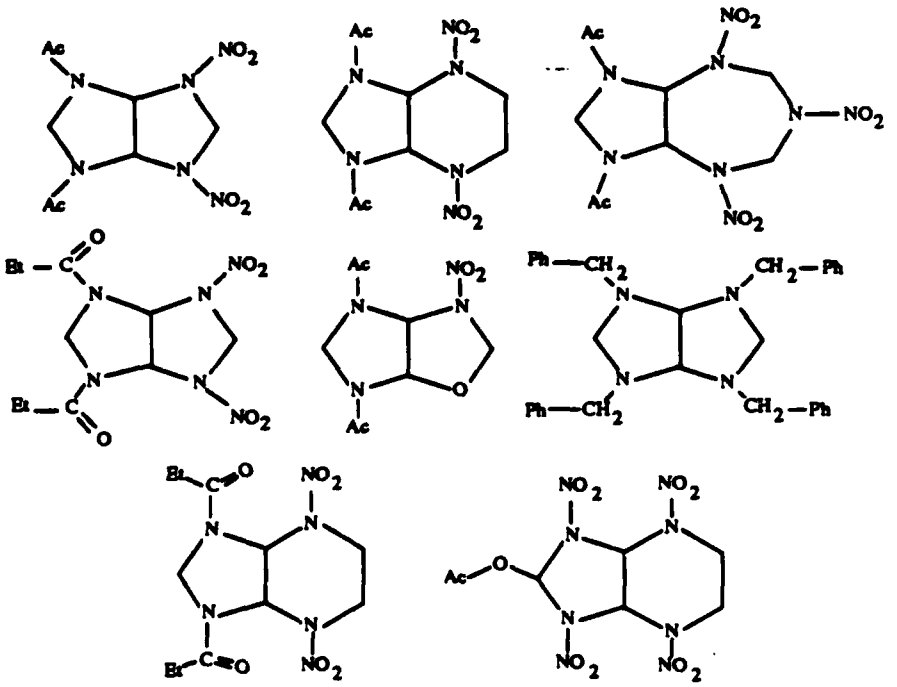


Abstract of a presentation given at the American Crystallographic Association Annual Meeting at Austin, Texas, March 1987.

STRUCTURE ANALYSIS OF ENERGETIC MATERIALS.

Judith L. Flippen-Anderson, Clifford F. George, and Richard Gilardi, Laboratory for the Structure of Matter, The Naval Research Laboratory, Washington, D.C. 20375-5000, USA

Much research effort goes towards the production of new materials which are dense, stable, and highly energetic. The density is quite important because the impulse provided by a propellant depends at least quadratically on its density. Today, HMX (2,4,6,8-tetranitro-2,4,6,8-tetraazacyclooctane) is the best known commercial material of this type. A fused-ring variant, "bicyclo-HMX", is predicted to be even better and it serves as a target molecule in several organic synthesis programs. Along the way, several unusual heterocycles have been discovered and, in several cases, identified through X-ray diffraction. We report here a number of these new compounds.



Abstract of a presentation given at the American Crystallographic Association Annual Meeting at Austin, Texas, March 1987.

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